

QV
25
K21s
1918

MANUAL OF
LABORATORY PRACTICE
FOR STUDENTS OF PHARMACY.

GEORGE REECHER KAUFFMAN, B. Sc., Phc. D.
JAMES HARTLEY BEAL, D. Sc., Phc. D.
JULIUS ARNOLD ROCH, Ph. D., Phc. D.



NLM 05070976 8

NATIONAL LIBRARY OF MEDICINE

SURGEON GENERAL'S OFFICE
LIBRARY.

ANNEX

Section

232804

No. 113,
W. D. S. G. O.

No. _____

3-513

1012

A SIMPLE COURSE

—OF—

Laboratory Practice in Applied Pharmacy

—BY—

✓
GEORGE BEECHER KAUFFMAN, B. SC., PHR. D.

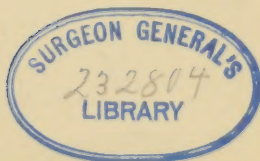
JAMES HARTLEY BEAL, D. SC., PHR. D.

JULIUS ARNOLD KOCH, PH. D., PHR. D.

COPYRIGHT, 1918

Third Edition

THE MIDLAND PUBLISHING COMPANY
COLUMBUS, OHIO



APR 14 1919
©CLAS15211

R

The use in this volume of certain portions of the text of the United States Pharmacopœia is by virtue of permission received from the Board of Trustees of the United States Pharmacopœial Convention. The said Board of Trustees is not responsible for any inaccuracy of quotation nor for any errors in the statement of quantities or percentage strengths.

QV
704
K215
1918

Table of Contents.

PART ONE—	
Physical Operations.....	4
PART TWO—	
Galenical Preparations	15
PART THREE—	
Preparation and Purification of Chemicals.....	31
PART FOUR—	
Prescription Practice.....	46
PART FIVE—	
Volumetric Analysis.....	56
PART SIX—	
Gravimetric Analysis.....	75
PART SEVEN—	
Pharmaceutical Assaying	83

PART ONE.

PHYSICAL OPERATIONS.

WEIGHTS AND MEASURES.

1. Practice in Metric Linear Measure.

1. With a meter stick take the dimensions of the laboratory in which you are working.

Draw a diagram in your notebook, showing the tables, doors, windows, and other fixed objects. Make the diagram on a scale of 2 centimeters to the meter.

Draw a second diagram of the laboratory and fixtures on a scale of $\frac{1}{4}$ inch to the foot, using a yardstick or foot rule for the measurements.

2. Compare the foot rule with the meter stick, and find the values of the inch, foot, and yard in centimeters, and of the inch in millimeters.

3. Ascertain and note your height, waist measure, length of arm and forearm in metric units. Also, express these values in inches.

NOTE.—Waist measure may be taken with a cord, and the cord then compared with the meter stick.

2. Comparison of Metric and Apothecaries' Fluid Measure.

1. Measure one fluidounce of water, pour into a cylinder, graduated in mils, and note the number of mils.

2. With a pipette graduated in mils, ascertain the number of mils in a fluidrachm.

3. Also determine the number of mils required to fill prescription vials holding respectively 2, 4, and 8 fluidounces. Do these bottles hold more or less than their names indicate?

4. With a measure graduated in minims, and a pipette graduated to mils, ascertain the number of minims in a mil.

5. Compare the results which you have obtained with the tables of equivalents published in the text books. If your results differ from the published equivalents, ascertain the probable reason for such difference.

3. Comparison of Metric and Apothecaries' Weight.

1. Note carefully the size and appearance of a gram weight; weigh it against and determine its value in grains.

2. Find the weight of the drachm and apothecaries' ounce in grams.

4. Practice in Weighing and Tying up Packages.

Weigh out, wrap, tie, and label such substances as sublimed sulphur, flax seed, copperas, pink root, senna, lycodium, etc., and submit the finished packages for the inspection of the instructor. Make the quantities of 2, 4, 6, and 8 avoirdupois ounces.

SPECIFIC GRAVITY.

5. Of Liquids.

Using apothecaries' weights, find the weight in grains of a clean, dry, ounce or half-ounce prescription vial. With a file, make a scratch on the neck of the vial, fill exactly to the mark with distilled water and again carefully weigh.

Empty the vial, and to dry it quickly rinse it out with a small quantity of alcohol and drain. When perfectly dry, fill the vial to the scratch with glycerin, and weigh. Make the calculations as follows:

1. Subtract the weight of the empty vial from the weight of the vial and water to find the weight of the water.

2. Subtract the weight of the vial from the weight of the vial and glycerin to obtain the weight of the glycerin.

3. Divide the weight of the glycerin by the weight of the water.

The result will be the specific gravity of the glycerin.

Repeat the foregoing experiment, using metric weights, and substituting alcohol for glycerin.

Repeat the experiments, using cotton seed oil, oil of wintergreen, water of ammonia, and solution of tersulphate of iron. Compare your results with the specific gravities given for the same substances in the Pharmacopœia.

NOTE.—When specific gravity is to be used as a criterion of quality, the determination must be made at the standard of temperature adopted by the Pharmacopœia, viz., 25°C.

If a regular specific gravity bottle is at hand this may be substituted for the prescription vial in the last experiments.

6. *Solids Heavier Than, and Insoluble in Water.*

1. Carefully weigh a small glass stopper, using either metric or apothecaries' weights, as preferred.

2. By means of a fine thread suspend the stopper from the hook at the end of the balance beam so that it will be completely immersed in water contained in a beaker resting on a small bench placed above the pan, and weigh the stopper as immersed.

Calculate as follows:

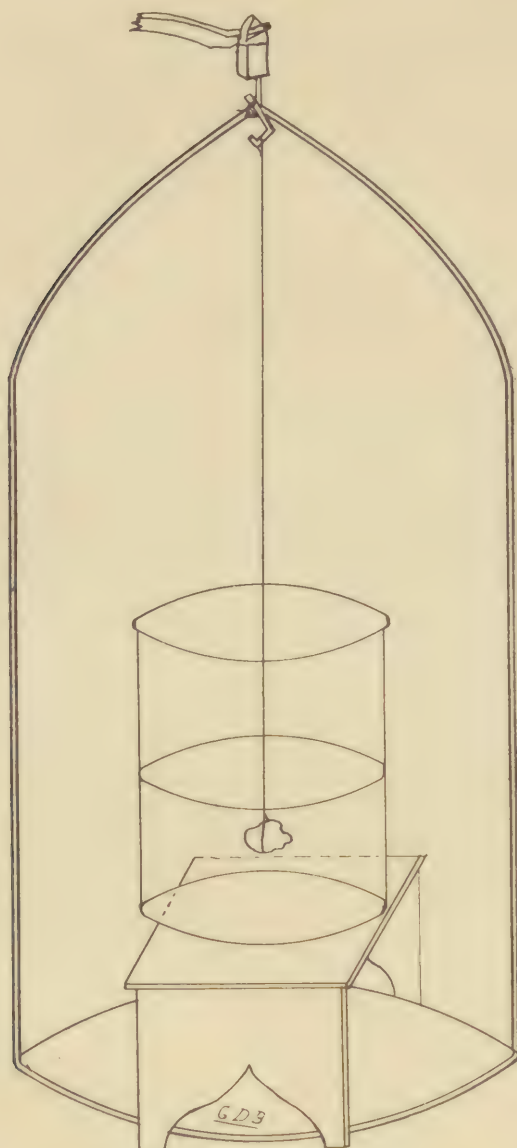
3. Subtract the weight of the stopper in water from its weight in air.

4. Divide the weight in air by the loss of weight in water.

NOTE.—The loss in weight is only apparent. The stopper is buoyed up by the weight of the water displaced.

The quotient will be the specific gravity of the glass.

Repeat the experiment with various solids which are heavier than, and insoluble in water.



Weighing a Solid in Water.

7. *Solids Lighter Than, and Insoluble in Water*

1. Tie a small lump of beeswax to the glass stopper used in the last experiment and take the weight of both in air, and then weigh the two together when immersed in water.

Make the calculation as follows:

2. From the combined weight of the glass stopper and wax in air subtract their weight when immersed in water, to find the loss of weight of both.

3. From the loss of weight of both, subtract the amount lost by the stopper when weighed in water alone, as determined by the preceding experiment. The difference is the weight lost by the wax when immersed.

4. Divide the weight of the wax in air by the loss of weight in water.

The result is the specific gravity of the wax.

8. *Solids Heavier Than, and Soluble in Water.*

Use a crystal of alum, blue vitriol, or citric acid, proceeding as in experiment 6, but immersing in benzin instead of water.

Make the calculation as follows:

1. Subtract the weight of the crystal when immersed in benzin from its weight in air.

2. Divide the weight in air by the loss of weight in benzin. This gives the specific gravity of the crystal compared to benzin.

3. Multiply the specific gravity thus found by the specific gravity of the benzin. The result is the specific gravity of the crystal as compared to water.

9. *Specific Gravity of Liquids Without the Use of a Bottle.*

1. Support a small glass stopper at the end of the balance beam and weigh first in air, then in distilled water.

2. Wipe the stopper and weigh it in the liquid the specific gravity of which is desired; for example, glycerin, sulphuric acid, or benzin.

Calculate as follows:

3. Subtract the weight in water from the weight in air to obtain the weight of a volume of water equal to the volume of the stopper.

4. Subtract the weight of the stopper when in the liquid under determination from its weight in air to obtain the weight of an equal volume of that liquid.

5. Divide the loss of weight of the stopper in the liquid under determination by its loss of weight in water.

The result is the specific gravity.

Repeat the experiment with various liquids suggested by the instructor.

10. Specific Gravity of a Solid in Powder Form.

1. Use the vial or specific gravity bottle employed in experiment No. 5, the weight of which has been accurately taken.

2. Fill the dry bottle about half full of manganese dioxide and carefully weigh. Next fill the bottle with distilled water, agitating so as to thoroughly wet the powder and remove all air bubbles, bring the liquid to the mark, and again weigh.

Calculate as follows:

3. From the weight of the vial and powder subtract the known weight of the vial to obtain the weight of the powder.

4. From the weight of the vial, powder and water subtract the weight of the vial and powder to find the weight of the water.

5. From the weight of water which the vial will hold, as determined in experiment 5, subtract the weight of water which it holds with the powder present, to find weight of water displaced by the powder.

6. Divide the weight of the powder by the weight of the water displaced.

The result will be the specific gravity.

11. Use of the Hydrometer.

1. Fill a tall hydrometer jar with water, and float in it two hydrometers, one for liquids heavier, and one for liquids lighter than water. Note the point to which each hydrometer sinks.

2. Empty the jar and fill with some heavier liquid, as a solution of common salt, solution of ammonium nitrate, etc., and determine its specific gravity with a hydrometer for heavy liquids.

3. Repeat the experiment with some liquid lighter than water, as alcohol, turpentine, etc., using the other instrument.

Note the temperatures of the liquids and record the results.

OPERATIONS INVOLVING THE USE OF HEAT.

12. Fall of Temperature Through Evaporation.

Rest a watch glass in a few drops of water on the table. Fill the watch glass with ether and with a blow-pipe or piece of glass tubing direct a jet of air obliquely against the surface of the ether, so as to produce rapid volatilization. When the ether has nearly all evaporated the watch glass will be found frozen to the table.

Record the result, and explain the phenomenon.

NOTE.—The evaporation must be rapid or the freezing will not take place.

13. Lowering of Temperature by Simple Solution.

Place in a beaker 200 mls of water, and take the temperature with a chemical thermometer. Add 50 gm. ammonium nitrate in crystals, and promote rapid solution by stirring with the thermometer.

Note the lowest point to which the thermometer falls, and express the reduction in temperature in both Centigrade and Fahrenheit degrees.

NOTE.—The solution thus obtained may be used for taking specific gravity by means of the Hydrometer (Experiment No. 11), after which the salt can be recovered in solid form by evaporation.

14. Sublimation of Benzoic Acid.

Crush to a coarse powder 20 gm. of benzoin, add an equal bulk of clean, dry sand, and place in a tinned iron or earthenware vessel of about one pint capacity.

Tie over the top of the vessel a piece of filter paper. Make a cone of stiff writing paper, like a "dunce cap," about 15 cm. high, and set it loosely over the filter paper. The lower end of the cone should be just large enough to cover the rim of the vessel.

Now heat the vessel on a sand bath rather strongly for about twenty minutes. When cold, remove the cone and shake out the crystals of benzoic acid.

Repeat the heating and cooling as long as crystals are obtained.

15. *Determination of Melting Point.*

Make a melting point tube by heating the center of a piece of heavy glass tubing (about 8 to 10 mm. diameter) in the Bunsen flame and drawing it out so as to produce tubing of one to two mm. diameter.

With a file cut this narrow tubing into pieces six to eight cm. long and close one end of each piece by holding in the edge of the flame.

Fill one of the tubes to the extent of about six mm. with the finely powdered and dry substance the melting point of which is to be determined.

Fit a melting point flask* with a cork perforated to fit your thermometer and notched at the side to permit of the escape of the air. Fill the flask with strong sulphuric acid to a height sufficient to cover the bulb of the thermometer completely, the lower end of the thermometer being about 6 mm. from the bottom of the flask.

On dipping the thermometer in the sulphuric acid, the melting point tube containing the substance of which the melting point is



Melting Point Determination.

* If a melting point flask is not at hand, use an ordinary chemical flask of thin glass.

to be determined, will adhere to the thermometer and the whole is to be immersed in the sulphuric acid and very slowly heated until the substance melts.

Note the temperature, which is the melting point. Allow the contents of the flask to cool and again heat carefully to verify your first results.

Use acetanilid, citric acid, and petrolatum as the subjects of the experiments.

16. Determination of Boiling Point.

Use the same flask used for the melting point determination, filling the bulb of the flask half full of the liquid, the boiling point of which is to be determined, and suspend the thermometer in the flask with its lower end about 2 cm. above the surface of the liquid.

Now boil steadily until the thermometer indicates a constant temperature, which note. Boil five minutes longer, during which time the thermometer should show no variation.

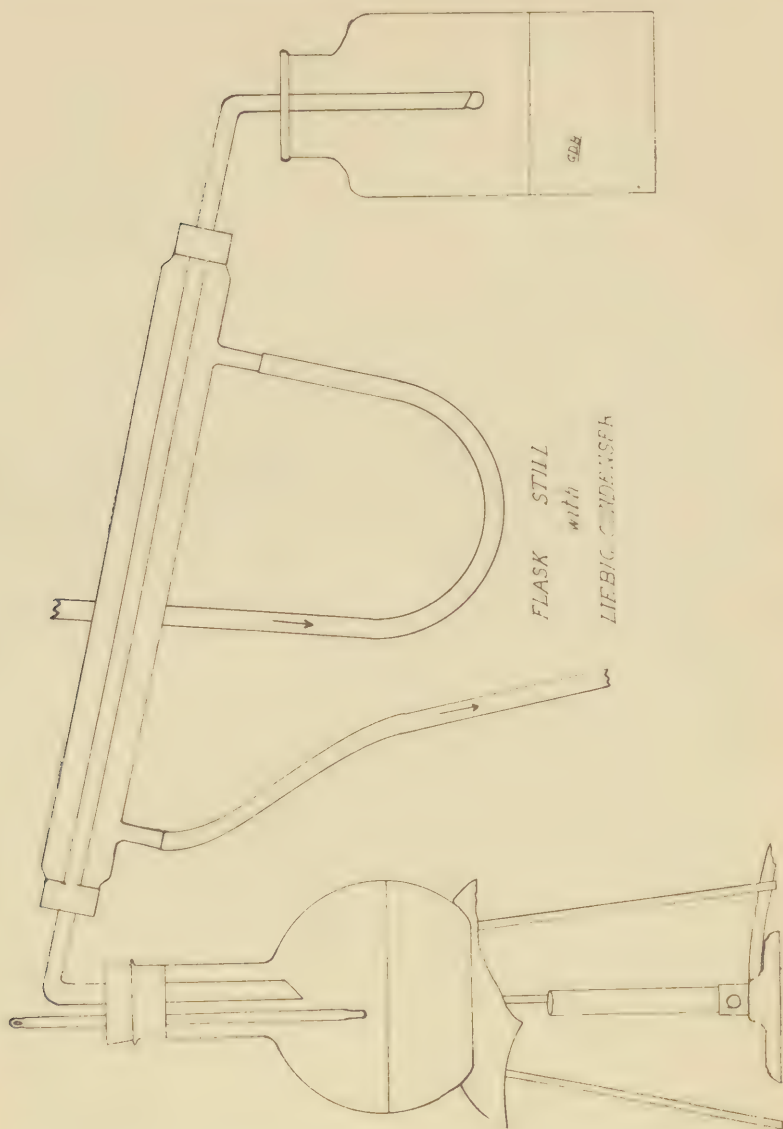
Use water, alcohol, chloroform, and solution of ferric chloride as the subjects of the experiments.

17. Flask Distillation.

Fit a 500 mil flask with a stopper having three holes. Place in the flask 200 mils of some alcoholic liquid (an old tincture or fluidextract) diluted with 100 mils of water.

Through one hole in the stopper pass a chemical thermometer so that the bulb is about 5 cm. above the surface of the liquid. Through another hole introduce a funnel tube reaching to within 2 cm. of the bottom of the flask. Through the remaining hole pass a bent tube, the outer end of which is connected with a Liebig condenser.

Attach the condenser to a source of water supply, support the flask on a piece of wire gauze, apply heat, and distill until 200 mils of distillate are collected in a graduated receiver.



The funnel tube may be omitted if a three-hole stopper is not at hand.

Note the temperature when the liquid begins to distill, and at the end of the operation.

Fill a specific gravity bottle with the distillate, take the specific gravity and consult the tables of the U. S. P. to learn the percentage strength of the alcohol. This will be the alcoholic strength of the original liquid.

The specific gravity may be determined at either 15.6° C. or 25° C.

NOTE.—The alcohol recovered may be preserved for use in future experiments.

PART TWO.

GALENICAL PREPARATIONS.

NOTE.—These experiments are given to illustrate galenical processes, and do not necessarily yield official products.

18. *Peppermint Water.*

Purified Talc 1.5 Gm.
Oil of Peppermint 0.2 Mil.
Distilled Water sufficient to make 100 Mils. ~

Place the purified talc in a mortar, add the oil of peppermint from a graduated pipette and mix. Add the distilled water gradually, with constant trituration, and filter through a well wetted filter. If necessary, return the filtrate to the filter until it runs clear.

19. *Rose Water.*

Fresh Rose Petals, any convenient quantity.
Water sufficient.

Tie the rose petals *loosely* in mosquito netting, and suspend the package in a still so that *it will not touch the sides or bottom*, add water and distill 100 mls or more of liquid. If oily particles float on top of the distillate, or if the latter is cloudy, filter through a well wetted filter.

If rose petals are not obtainable, use any herb containing a volatile oil, as spearmint.

When the still is fitted with a wire basket this may be employed for holding the herb instead of the mosquito netting.

20. *Solution of Potassium Arsenite.*

(Fowler's Solution.)

Arsenic Trioxide in fine powder 1 Gm.
Potassium Bicarbonate 2 Gm.
Compound Tincture of Lavender 3 Gm.
Distilled Water sufficient to make 100 Gm.

Boil the arsenic trioxide and potassium bicarbonate with 50 gm. of distilled water, in a tared capsule, until dissolved, and add sufficient distilled water to make the solution weigh 97 gm. when cold.

Lastly, add the compound tincture of lavender, and after standing twenty-four hours, filter through paper.

21. Solution of Lead Subacetate.

Lead Acetate.....	18 Gm.
Lead Oxide.....	11 Gm.
Distilled Water sufficient to make 100 Gm.	

Dissolve the lead acetate in 70 mls boiling distilled water contained in a glass beaker or porcelain dish, and add this solution to the lead oxide, finely powdered, and boil for half an hour, adding distilled water as necessary, to make up the loss by evaporation. Remove the vessel from the source of heat, and when cold, add sufficient distilled water, previously boiled and cooled, to make the solution weigh 100 gm. Lastly, filter the liquid through filter paper in a closely covered funnel.

22. Decoction of Eupatorium.

Eupatorium Herb, closely broken.....	5 Gm.
Water sufficient to make 100 Mils.	

Tie the eupatorium loosely in a piece of cheese cloth or thin muslin, and by means of a string suspend in an earthen teapot or other vessel so that it will not touch the side or bottom. Add 100 mls water and boil for fifteen minutes. Cool, express, and add sufficient water through the strainer to make 100 mls.

To sterilize the decoction, place it in a clean bottle, cork loosely and stand the bottle in a vessel of cold water and heat gradually to boiling. After boiling about five minutes, remove the bottle, press the cork in tightly and tie it down with a string.

23. Infusion of Chamomile.

Anthemis	5 Gm.
Boiling water sufficient to make 100 Mils.	

Tie the anthemis loosely in a piece of cheese cloth or muslin, suspend in an earthen vessel, pour on 100 mls boiling water, cover the vessel and let stand till cool. Lastly, pour off the cooled liquid, and add through the drug suspended in a funnel, sufficient water to make 100 mls.

To preserve, sterilize by heat, as directed under Decoc-tion of Eupatorium.

24. *Syrup.*

(Simple Syrup.)

Granulated Sugar..... 85 Gm.
Distilled Water sufficient to make 100 Mls.

Place the sugar and 45 mls of distilled water in a porcelain dish or tinned iron pan, raise the temperature to boiling and when the sugar is completely dissolved, strain through muslin. Lastly, pour sufficient distilled water through the strainer to make the liquid measure 100 mls and mix thoroughly.

Preserve in well-corked bottles for use in subsequent preparations.

25. *Syrup of Wild Cherry.*

Wild Cherry Bark in No. 20 Powder.... 15 Gm.
Granulated Sugar 80 Gm.
Glycerin 5 Mils.
Distilled Water sufficient to make 100 Mls.

Mix the glycerin with 20 mls of water, moisten the wild cherry bark with 10 mls of this mixture, pack firmly in a cylindrical percolator, and pour the remaining menstruum upon it. When the liquid has disappeared from the surface of the drug, add sufficient water to saturate the drug and leave a stratum above it. Close the lower orifice, cover the percolator, allow the drug to macerate 24 hours. Allow the percolation to proceed slowly, using water as a menstruum, until 50 mls of percolate have been obtained. In this dissolve the sugar by agitation without heat and add enough water to make the product measure 100 mls. Lastly strain the product.

26. *Syrup of Ferrous Iodide.*

Fine Iron Wire, free from rust 1.25 Gm.
Iodine 4.15 Gm.
Dil. Hypophosphorous Acid..... 2 Mils.
Sugar 60 Gm.
Distilled Water sufficient to make 100 Gm.

Cut the wire into small pieces and place in a flask of thin glass, add 15 mils of distilled water, and then the iodine. Shake the flask occasionally until the solution has acquired a greenish color and has *lost the odor of iodine*.

Heat the solution to boiling and filter through a double filter into the sugar contained in a tared vessel. **Rinse the flask** with 12.5 mils of distilled water, and pass through the filter.

Lastly, heat the mixture on a water-bath until solution is effected, strain into a tared bottle, and add the hypophosphorous acid and enough distilled water to make 100 gm.

27. *Compound Mixture of Glycyrrhiza.*

(Brown Mixture.)

Pure Extract of Glycyrrhiza	3	Gm.
Syrup	5	Mils.
Granulated Acacia	3	Gm.
Camphorated Tincture of Opium	12	Mils.
Antimony and Potassium Tartrate ...	0.024	Gm.
Spirit of Nitrous Ether	3	Mils.
Water sufficient to make 100 Mils.		

Rub the extract of glycyrrhiza and acacia in a mortar with 50 mils of water till dissolved, and transfer to a graduated vessel containing the other ingredients, add the antimony and potassium tartrate, dissolved in 2 mils of hot water. Rinse the mortar with sufficient water to make 100 mils and add to the mixture. Mix by shaking.

28. *Tincture of Arnica.*

Arnica Flowers in No. 20 Powder.....	20 Gm.
Diluted Alcohol sufficient to make 100 Mils.	

Macerate the arnica with 50 mils of diluted alcohol in a closed vessel in a warm place for three days, with occasional stirring, and express strongly.

Repeat the operation twice successively with 25 mils diluted alcohol, macerating twenty-four hours each time. Mix the expressed liquids, note the volume and macerate the marc with sufficient diluted alcohol to make approximately 100 mils and express as before.

Filter the mixed liquids and pass sufficient diluted alcohol through the filter to make 100 mils.

29. *Camphorated Tincture of Opium.*

(Paregoric.)

Powdered Opium	0.4 Gm.
Benzoic Acid	0.4 Gm.
Camphor	0.4 Gm.
Oil of Anise	0.4 Mil.
Glycerin	4 Mils.
Diluted Alcohol.....	95 Mils.

Place the opium, benzoic acid, camphor, oil of anise, and glycerin in a flask and add the diluted alcohol. Macerate for three days, with frequent shaking. Filter through paper, in a covered funnel, and pass sufficient diluted alcohol through the filter to make 100 mils.

30. *Fluidextract of Sarsaparilla.*

Sarsaparilla, in No. 30 Powder.....	100 Gm.
Alcohol,	
Water, of each, sufficient to make	100 Mils.

Fit a narrow percolator with a control tube as shown in the illustration, and place a tuft of purified cotton in the bottom to prevent the drug from closing the entrance to the tube.

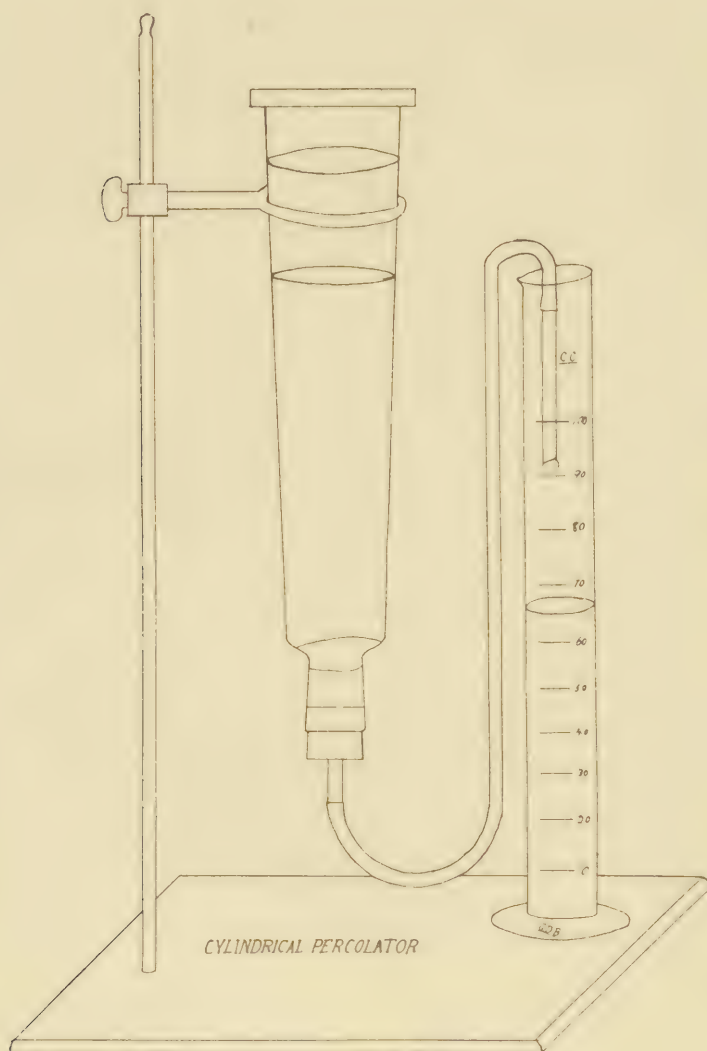
Prepare the menstruum by mixing one volume of alcohol with two volumes of water.

Moisten the drug with 40 mils of the menstruum, pack firmly in the percolator, and cover the surface of the drug with a piece of filter paper cut to fit. Place a glass stopper or quartz pebble on top of the paper to prevent it from floating when the menstruum is poured in.

Pour on menstruum until the liquid begins to drop from the percolator and there is a stratum of liquid on top of the drug.

Raise the end of the control tube so as to stop the flow, closely cover the percolator, and macerate 48 hours.

Lower the tube and allow percolation to proceed at the rate of 2 to 5 drops per minute until the drug is exhausted.



Percolator fitted with Control Tube.

Collect and reserve the first 80 mils of the precolate separately.

Evaporate the remaining weak percolate to the consistence of a soft extract, dissolve in the reserved percolate, and add sufficient menstruum to make 100 mils.

31. Fluidextract of Triticum.

Triticum, finely cut 100 Gm.

Alcohol,

Water, each, a sufficient quantity to make 100 Mils.

Prepare a cylindrical glass percolator in the usual manner, with cotton, and perforated cork having a glass tube, but omitting the rubber control tube.

Pack the dry drug in the percolator, wrap the percolator from top to bottom in a towel wrung out of hot water, and pour boiling water on the drug, carefully added at first until the percolator is thoroughly heated.

Continue the percolation until the drug is exhausted.

Evaporate the percolate to 75 mils, add 25 mils alcohol, and set aside for 48 hours.

Filter the liquid and add sufficient of a mixture of one volume of alcohol and three volumes of water to make 100 mils.

NOTE.—The Pharmacopœia directs the use of a metallic percolator, because of the danger of fracturing a glass percolator through use of hot water.

By using the hot towel and adding the hot water carefully at first, as above directed, this danger may be avoided. The percolator should, of course, be kept hot during the entire operation.

32. Fluidextract of Eupatorium.

Eupatorium, in No. 40 Powder. 100 Gm.

Diluted Alcohol sufficient to make 100 Mils.

Moisten the powder with 40 mils of diluted alcohol, pack firmly in a narrow, cylindrical percolator, fitted with control tube, cover the top of the powder with a piece of filter paper neatly fitted to the percolator, and add sufficient diluted alcohol to saturate the powder and leave a stratum above it.

When the liquid begins to drop from the percolator, raise the control tube so as to stop the flow, closely cover the top of the percolator to prevent evaporation and allow to macerate for 48 hours.

Then lower the tube and allow percolation to proceed, adding diluted alcohol as necessary, until 80 mils of percolate are obtained. Set this percolate aside and continue the percolation until the drug is exhausted.

Evaporate the weak percolate on a water-bath until it reaches the consistence of a soft extract. Dissolve this in the reserved 80 mils of percolate, and add sufficient diluted alcohol to make the product measure 100 mils.

If a precipitate forms on standing, filter through paper.

The percolation should not proceed at a greater rate than 2 to 5 drops per minute, which rate may be obtained by raising or lowering the end of the control tube.

Before beginning the percolation, a four-ounce bottle should have a piece of paper pasted on the side to show the point at which it will contain 80 mils. This bottle is used as a receiver for the first percolate.

33. *Fluidextract of Wild Cherry.*

Wild Cherry, in No. 30 Powder..... 100 Gm.
Glycerin 20 Mils.
Alcohol and Water, each, a sufficient quantity to
make 100 Mils.

Mix the glycerin with 20 mils of alcohol and 60 mils of water, and moisten the powder with 30 mils of this mixture.

Pack firmly in a cylindrical percolator, and add menstruum sufficient to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close and cover the percolator and macerate for 48 hours.

Allow percolation to proceed, gradually adding first the remainder of the menstruum and afterward a mixture of alcohol and water in the proportion of two volumes of alcohol to four volumes of water, and allow the percolation to continue very slowly until the percolate measures 100 mils.

34. Wine of Antimony.

Antimony and Potassium Tartrate....	0.4 Gm.
Boiling Distilled Water	2.5 Mils.
Sherry Wine sufficient to make 100 Mils.	

Dissolve the antimony and potassium tartrate in the boiling, distilled water, and add the solution to 72.5 mls of sherry wine. Mix well and allow to stand until cool. Filter through paper and add sufficient sherry wine through the filter to make 100 mls.

35. Glycerite of Starch.

Starch	2.5 Gm.
Glycerin	20 Gm.
Distilled Water.....	2.5 Mils.

Triturate the starch with the water to a smooth mixture, then gradually add this to the glycerin contained in a porcelain dish and heat to about 140° C., stirring constantly at that temperature until a translucent jelly is produced. Transfer to a wide-mouthed vial, as a morphine vial, and stopper closely.

The heat must not be allowed to rise above 140° C., and the mixture must be constantly stirred, or the jelly will be discolored.

36. Compound Powder of Glycyrrhiza.

Senna, in No. 30 Powder.....	18 Gm.
Glycyrrhiza, in No. 80 Powder.....	23.6 Gm.
Washed Sulphur.....	8 Gm.
Sugar, in Fine Powder	50 Gm.
Oil of Fennel	0.4 Gm.

Add the oil of fennel to 25 gm. of sugar contained in a mortar and triturate, add 25 gm. of sugar and again triturate. Then add the remaining ingredients and triturate all together. Finally, pass the powder through a No. 80 sieve. Keep in a well-stoppered bottle.

NOTE.—In triturating, move the pestle in a spiral from the center to the circumference, and then from the circumference to the center. Scrape the sides of the mortar frequently with a flexible spatula.

37. Extract of Malt.

Malt, coarsely ground..... 100 Gm.
Water, a sufficient quantity.

Place the malt in a vessel of about 800 mls capacity, add 100 mls water, mix well, cover and macerate for six hours.

Add 400 cc. water heated to about 30° C., mix and digest one hour on a water-bath at a temperature not above 55° C.

Strain through muslin with strong expression, and pass the strained liquid through a tuft of cotton loosely pressed into the neck of a funnel.

Evaporate the liquid (preferably on a water-bath), at a temperature not above 55° C., to the consistence of thick honey.

Keep in a well closed vessel in a cool place.

38. Pure Extract of Glycyrrhiza.

Glycyrrhiza, in No. 20 Powder..... 100 Gm.
Ammonia Water..... 15 Mils.
Glycerin and Water, each, a sufficient quantity.

Mix the ammonia water with 300 mls of water, moisten the drug with 100 mls of the mixture and macerate in a closed vessel for 24 hours.

Pack moderately in a glass percolator, and add gradually, first the remainder of the menstruum and then water until the drug is exhausted.

Lastly, evaporate the percolate in a tared dish on a water-bath to a pilular consistence, and while it is still warm incorporate with it five per cent of its weight of glycerin.

If the extract is desired in the form of a powder, the evaporation should be continued until nearly dry, leaving out the glycerin, then removed from the dish by means of a spatula, and when cold, powdered in a mortar.

NOTE.—The process may be hastened by evaporating the percolate over a naked flame or on a sand-bath until reduced to about one-third or one-fourth of its volume, and the process completed on a water-bath, but the product will not be of as good a quality as if evaporated entirely on the water-bath.

39. Purified Aloes.

Aloes	50 Gm.
Alcohol	10 Mils.

Heat the aloes on a water-bath till completely melted. Add the alcohol, mix thoroughly, and strain through a No. 60 sieve just previously dipped in boiling water. Evaporate the strained liquid on a water-bath with constant stirring, until a thread of the mass is brittle on cooling. When cold, break the mass into small pieces and preserve in a well-stoppered bottle.

To be used in subsequent preparations.

40. Pills of Aloes.

Purified Aloes, in Fine Powder.....	6.5 Gm.
Soap, in Fine Powder	6.5 Gm.
Water, a sufficient quantity.	

Thoroughly mix the powders in a small mortar, add water, drop by drop, kneading thoroughly with each addition, until a plastic mass is produced.

Transfer to a pill tile, sprinkle with powdered glycyrrhiza, and roll into a thin pencil, long enough to cover fifty divisions of the graduated space on the tile.

With the spatula, square up the ends of the cylinder and carefully cut into fifty equal portions. Sprinkle some glycyrrhiza in the palm of the left hand, take one of the divisions at a time and with the first two fingers of the right hand roll the pill until perfectly spherical.

Lastly, place in a pill box with a small amount of powdered glycyrrhiza.

Only the powdered Russian licorice should be used, and this only in small quantity.

41. Compound Pills of Rhubarb.

Rhubarb, in No. 60 Powder.....	3.25 Gm.
Purified Aloes, in Fine Powder.....	2.5 Gm.
Myrrh, in Fine Powder.....	1.5 Gm.
Oil of Peppermint.....	0.25 Mil.

Place the powders in a mortar, add the oil of peppermint, and mix thoroughly. Make a mass by the addition of water, drop by drop, kneading thoroughly after each addition.

Roll into a pencil, divide into twenty-five equal portions, and finish as described under Pills of Aloes.

42. *Pills of Ferrous Carbonate.*

(Blaud's Pills.)

Granulated Ferrous Sulphate	8	Gm.
Potassium Carbonate	4	Gm.
Sugar	2	Gm.
Tragacanth, in Fine Powder	0.50	Gm.
Althæa, in No. 60 Powder	0.50	Gm.
Glycerin, Water, of each, sufficient to make a mass.		

Rub the potassium carbonate in a mortar with about 6 drops of glycerin and water until reduced to a smooth mixture.

Triturate the ferrous sulphate and sugar to a uniform powder, add to the potassium carbonate, and work the mass thoroughly until it assumes a greenish color, and effervescence has ceased.

Incorporate the tragacanth and althæa, adding water a drop at a time, if necessary, and when a suitable mass has been obtained, divide into fifty parts, and finish as directed under Pills of Aloes.

43. *Pills of Phosphorus.*

Phosphorus	0.03	Gm.
Althæa, in Fine Powder	3.00	Gm.
Acacia, in Fine Powder	1.5	Gm.
Chloroform, Glycerin, Water, Balsam of Tolu, Ether, of each, a sufficient quantity.		

Dissolve the phosphorus in a test tube in about 3 mils of chloroform, with the aid of a gentle heat, replacing the chloroform lost by evaporation.

Mix the powders in a mortar, add the solution of phosphorus, and add immediately about 1.25 mils of glycerin and $\frac{3}{4}$ mil of water.

Mass quickly and form into fifty pills in the usual manner.

Dissolve 5 gm. of dry, hard balsam of tolu in 8 mils of ether, rotate the pills in this solution, contained in a small evaporating dish, until uniformly coated, and transfer to a plate or pill tile to dry, rolling them about to prevent their sticking together.

NOTE.—Where a number of students are to make the pills of phosphorus, it will be more convenient for the instructor to make up a sufficient quantity of the chloroform solution for the whole class.

44. *Troches of Cubeb*

Oleoresin of Cubeb	1 Gm.
Oil of Sassafras.....	0.5 Mil.
Extract of Glycyrrhiza, in Fine Powder.	12.5 Gm.
Acacia, in Fine Powder.....	6 Gm.
Syrup of Tolu, a sufficient quantity.	

Triturate the powders together until thoroughly mixed. Incorporate the oleoresin and oil, and with syrup of tolu form a mass.

Sprinkle a troche board, or a pill tile, with powdered sugar, and on this roll the mass into a flat sheet. Cut into 50 troches with a troche cutter, or if a troche cutter is not at hand, square the edges of the mass with a spatula, and cut into 50 equal-sided squares.

Care must be taken to roll the mass to just such a thinness that when cut it will make exactly 50 troches, after working up the scraps, and no more.

Another method is to roll the mass into a thick cylinder, as if it were to be made into large pills or boli, divide into 50 equal divisions, roll each division into a bolus and then flatten into a troche with a spatula. Or place the bolus in a troche cutter made of brass tubing, and flatten in the tube by use of a plunger, and afterwards eject.

45. *Extract of Gentian.*

Gentian, in No. 20 Powder.....	100 Gm.
Water, sufficient.	

Moisten the powder with 40 mils of water and macerate for 24 hours. Pack in a conical percolator and percolate with

water until practically exhausted. Reduce the liquid to three-fourths of its bulk by boiling, and then continue the evaporation on a water-bath until the product is reduced to a pilular consistence.

46. Oleoresin of Cubeb.

Cubeb, in No. 30 Powder..... 100 Gm.

Alcohol, sufficient.

Pack the powder firmly in a cylindrical glass percolator, and percolate slowly with alcohol until the drug is exhausted.

Recover the greater portion of the alcohol by distillation on a water-bath (see "Flask Distillation" in Part One), transfer the residue to a shallow dish, in a warm place, and allow the remaining alcohol to evaporate with occasional stirring.

Preserve in a well-stoppered bottle.

On standing for some time, a waxy, crystalline matter separates, which should be rejected.

47. Resin of Podophyllum.

Podophyllum, in No. 60 Powder..... 100 Gm.

Hydrochloric Acid..... 10 Mils.

Alcohol, Water, of each, sufficient.

Moisten the powder with 48 mils of alcohol, pack firmly in a narrow cylindrical percolator, and add enough alcohol to saturate the powder and leave a stratum above. When the liquid begins to drop from the percolator, close the lower orifice, cover the percolator and macerate for 48 hours.

Allow percolation to proceed until 160 mils of percolate are obtained, or until the percolate ceases to produce more than a slight turbidity when dropped into water.

Distill off the alcohol by means of a water-bath until the percolate is reduced to a thin syrup and pour it slowly, with constant stirring into 200 mils of water cooled to 10° C. or below, and containing the hydrochloric acid.

When the precipitate has subsided, decant the supernatant liquid and wash the precipitate twice by decantation with cold water.

Lastly, spread the resin on a strainer and dry in a cool, dark place.

Should the resin become lumpy on drying, rub it to powder in a mortar.

48. *Ointment.*

(Simple Ointment.)

White Wax	20 Gm.
Benzoinated Lard.....	80 Gm.

Melt the wax, add the benzoinated lard, and when liquefied, remove from the source of heat, and stir the mixture gently until it cools.

49. *Soft Soap.*

Linseed Oil	100 Gm.
Potassa	24 Gm.
Alcohol	10 Mils.
Water, a sufficient quantity.	

Heat the linseed oil in a deep, capacious vessel on a water-bath, to a temperature of about 70° C.

Dissolve the potassa in 112.5 mils of water, heat the solution to about 70° C. and add, with constant stirring, to the linseed oil.

Incorporate the alcohol and continue the heat without stirring until a portion of the mixture is found to be soluble in boiling water without the separation of oily drops. Allow the mixture to cool and transfer to suitable vessels.

NOTE.—The potassa used in this formula should be of full official strength. Potassa of less strength may be used if a proportionately greater amount be taken.

50. *Ointment of Mercuric Nitrate.*

(Citrine Ointment.)

Place 76 gm. of lard, free from water and salt, in a glass or porcelain vessel and heat to 105° C. Withdraw the heat, and add gradually 7 gm. nitric acid.

When the reaction has moderated, re-apply heat until

effervescence ceases. When the mixture has cooled to 40° C., dissolve 7 gm. mercury in 10.5 gm. nitric acid with the aid of sufficient heat to prevent crystallizing, and add it to the mixture. When the mass begins to solidify, mix thoroughly with a wooden spatula. Avoid contact with metal. Preserve in a well-closed vessel in a cool, dark place.

51. *Lard.*

Procure about 200 gm. of "leaf lard" at the butcher's, cut off adherent non-fatty tissue, wash free from blood and dirt, and wipe dry with a clean towel.

Cut the lard into small pieces, place in an enamel or porcelain dish and heat on a water-bath, frequently pressing the particles of tissue against the sides of the dish, by means of a pestle or piece of hard wood, to hasten the separation of the liquid fat.

Pour off the melted lard from time to time through a muslin strainer, and when no more fat separates, transfer the whole to the strainer and express.

Heat the strained lard in a porcelain dish until it ceases to lose weight.

Preserve in well closed vessels in a cool place.

52. *Benzoinated Lard.*

Procure at the market about 150 gm. of leaf lard. Cleanse from dirt and blood, if necessary, by washing, and wipe dry with a clean cloth.

Cut the lard into small pieces, and place in a dish over a water-bath, and apply heat.

Coarsely powder 2 gm. benzoin, tie in a piece of muslin and immerse the bag in the lard. Cover the vessel and continue the heat, with occasional stirring, for two hours.

Strain through clean, dry muslin, and preserve in a well-closed vessel in a cool, dark place.

PART THREE.

PREPARATION AND PURIFICATION OF CHEMICALS.

53. *Purification and Granulation of Ammonium Chloride.*

Crude Sal Ammoniac.....	30 Gm.
Ammonia Water	5 Mils.
Distilled Water	100 Mils.

Dissolve the sal ammoniac in the water with the aid of heat. Add the ammonia water, filter through a fluted filter, and evaporate.

As soon as crystals commence to form, begin stirring and continue until the mass is dry and granular.

54. *Purification of Ferrous Sulphate.*

Crude Ferrous Sulphate, or Copperas..	50 Gm.
Distilled Water.....	80 Mils.
Iron Wire, in small pieces.....	2 Gm.
Sulphuric Acid, about	2.5 Mils.

Dissolve the ferrous sulphate in the water with the aid of heat. Add the iron wire and sulphuric acid, evaporate to about 100 gm. and filter.

Cover the dish and set aside to crystallize. Remove the crystals and dry on blotting paper. When dry, preserve in a well-stoppered bottle.

Further concentration of the mother liquor will produce a second crop of crystals.

55. *Purification of Sulphur.*

(Washed Sulphur.)

Sublimed Sulphur.....	25 Gm.
Ammonia Water.....	2.5 Mils.
Distilled Water.....	25 Mils.

Triturate the sulphur in a mortar with one-half of the water and all of the ammonia water, and transfer to a beaker. Wash out the mortar with the remainder of the water, and add to the mixture. Cover the beaker with a glass plate and let stand for 12 hours.

Convey the sulphur to a plain filter with the aid of water, wash until free from the odor of ammonia, and dry.

56. *Potassium and Sodium Tartrate.*

(Rochelle Salt.)

Sodium Carbonate.....	12 Gm.
Potassium Bitartrate.....	16 Gm.
Distilled Water	80 Mils.

Add the sodium carbonate to the water contained in a porcelain dish, heat to boiling, and gradually add the potassium bitartrate. When dissolved, cool and filter the solution, evaporate until a pellicle begins to form on the surface and set aside to crystallize.

After 24 hours pour off the mother liquor, drain, and dry the crystals on blotting paper.

57. *Red Mercuric Iodide.*

Corrosive Chloride of Mercury	8 Gm.
Potassium Iodide	10 Gm.

Dissolve each of the salts in 160 mls of distilled water. Filter the solutions separately. Pour the liquids simultaneously in a thin stream into a vessel containing 400 mls of distilled water. An assistant should stir the mixture constantly while the liquids are being poured.

Allow the precipitate to subside, and decant the supernatant liquid. Pour the precipitate upon a plain filter and wash with distilled water until the washings cease to give more than a faint cloudiness with solution of silver nitrate.

When the precipitate has drained, enclose the filter between sheets of blotting paper and dry in a dark place, at a temperature not exceeding 40° C.

Lastly, keep in tightly closed bottles protected from the light.

58. *Sodium Salicylate.*

Salicylic Acid.....	10 Gm.
Sodium Bicarbonate	6 Gm.
Distilled Water.....	60 Mils.

Triturate the salicylic acid in a mortar with one-half the water gradually added until a smooth mixture is formed.

Dissolve the sodium bicarbonate in the remainder of the water and add to the salicylic acid, in portions, with constant stirring, until effervescence ceases.

Filter through white filter paper and evaporate on a water-bath, with constant stirring until dry.

CAUTION.—Use distilled water and carefully avoid all contact with iron. If the smallest contamination with iron is permitted, the salt will have a pink or red color.

59. *Solution of Potassa.*

Potassium Bicarbonate.....	8.5 Gm.
Lime	4 Gm.
Distilled Water sufficient to make	100 Mils.

Dissolve the potassium bicarbonate in 40 mils of distilled water and heat slowly to boiling.

Slake the lime with about 2 mils of distilled water, add 40 mils of distilled water, and pour it into a tared flask of thin glass and heat to boiling.

To the boiling milk of lime gradually add the solution of potassium bicarbonate and boil for ten minutes.

Cool and weigh, and add sufficient distilled water to make the liquid weigh 100 gm. Strain the liquid through linen, and when it has settled, pour off the clear solution.

60. *Diluted Hypophosphorous Acid.*

Potassium Hypophosphite	4.16 Gm.
Tartaric Acid.....	6 Gm.
Distilled Water.....	12 Mils.
Diluted Alcohol.....	12 Mils.

Dissolve the potassium hypophosphite in the distilled water, and the tartaric acid in the diluted alcohol. Mix the two solutions in a flask, stopper and set aside in a cool place for 12 hours.

Filter through a funnel, the neck of which contains a small pellet of absorbent cotton. Transfer the liquid to an evaporating dish, the weight of which is known, weigh the dish and liquid and evaporate on a water-bath until it no longer smells of alcohol.

Cool, place the dish on the balance and add enough distilled water to restore the original weight of filtrate. Lastly, keep in well-stoppered bottles.

If it is desired to preserve the potassium bitartrate formed in this reaction, wash the crystals on the cotton with a small quantity of diluted alcohol, dissolve with the aid of hot water, evaporate the solution until a pellicle begins to form, and set aside to crystallize. Lastly, drain the crystals and dry between blotting paper.

61. *Antimony and Potassium Tartrate.*

(Tartar Emetic.)

Oxide of Antimony, in fine powder....	4 Gm.
Potassium Bitartrate	5 Gm.
Distilled Water.....	20 Mils.

Heat the water to boiling in a glass or porcelain vessel, mix the powders, add them to the boiling water, and boil for one hour, adding distilled water as necessary to replace loss by evaporation.

Filter while still moderately hot, and set aside for crystals to form. Remove the crystals and dry them.

Again evaporate the mother liquor to obtain a fresh crop of crystals. Purify the last crop of crystals by re-dissolving them in distilled water, and again crystallizing.

Lastly, reduce all the crystals to fine powder and preserve in a well-stoppered bottle. Mark with a poison label.

62. *Zinc Acetate.*

Oxide of Zinc	6.5 Gm.
Acetic Acid	25.5 Mils.
Distilled Water	15 Mils

Mix the acetic acid and water in a porcelain dish, add the oxide of zinc, and apply a gentle heat for half an hour, adding water as necessary to replace the loss by evaporation. Then raise the temperature to the boiling point, filter the solution while hot and set aside to crystallize.

Drain the crystals and place them on bibulous paper to dry. Slightly acidulate the mother liquor with acetic acid, evaporate to one-half, and set aside to obtain a second crop of crystals.

63. *Zinc Oleo-Palmitate.*

Powdered Castile Soap (Sodium Oleo-Palmitate)	5 Gm.
Zinc Acetate	2.5 Gm.

Dissolve the zinc acetate in about 200 mils of water, and filter through paper. Dissolve the soap in about 500 mils of water, using heat to hasten the solution, and filter through cotton. Pour the soap solution into the zinc acetate solution with constant stirring. Transfer the precipitate to a plain filter, wash thoroughly, dry and preserve in a well-stoppered bottle.

64. *Solution of Ferric Chloride.*

Fine Iron Wire, free from rust.....	12.5 Gm.
Hydrochloric Acid	68 Gm.
Nitric Acid,	
Distilled Water, of each, sufficient to make 100 Gm.	

Cut the wire into small pieces and place in a flask of thin glass. Add a mixture of 42 gm. hydrochloric acid and 25 mils distilled water, and let stand in a warm place until effervescence ceases.

Heat the liquid to the boiling point, filter through paper,

and rinse the flask and filter with a little hot distilled water. To the filtrate add 22.5 gm. hydrochloric acid, and pour the mixture slowly in a thin stream into 6.5 gm. nitric acid contained in a capacious porcelain dish.

When effervescence ceases, heat the dish on a sand-bath till free from nitrous odor. Remove a few drops of the solution, dilute with water, and add a few drops of freshly prepared solution of potassium ferricyanide. (Made by dissolving a crystal of the salt in distilled water in a test tube.)

Should the test produce a blue color, the iron has not been completely oxidized to ferric salt. In this case, add a little nitric acid, drop by drop, to the original solution as long as any effervescence can be noticed, and again heat the dish until the liquid ceases to give off nitrous fumes.

Finally to the solution add the remaining 4 gm. hydrochloric acid and sufficient distilled water to make the solution weigh 100 gm. If the liquid is cloudy, filter.

65. *Solution of Tersulphate of Iron.*

Ferrous Sulphate	50 Gm.
Sulphuric Acid	9.6 Gm.
Nitric Acid,	
Water, of each, a sufficient quantity.	

Coarsely powder the sulphate of iron and divide into four portions.

To 25 mls *water previously placed in an evaporating dish* add the sulphuric acid, heat to nearly 100° C. and add 5.6 gm. nitric acid.

Add the ferrous sulphate, a portion at a time, stirring after each addition until effervescence ceases. When all of the ferrous sulphate has been dissolved, add a few drops of nitric acid and boil the solution until it assumes a clear, reddish-brown color.

Remove a few drops of the solution, dilute with water, and test for unoxidized ferrous salt by adding a few drops of a freshly prepared solution of potassium ferricyanide. (Made by dissolving a crystal of the salt in a test tube with distilled

water.) If a blue precipitate is produced, add a few drops of nitric acid to the mixture in the dish, and again boil.

To test for the presence of nitric acid, remove a few drops of the solution, dilute with water, add an equal volume of concentrated sulphuric acid and a crystal of ferrous sulphate. If the crystal becomes surrounded with a brownish zone, the mixture contains free nitric acid, and should be boiled until it fails to show the reaction.

When the tests show the absence of both ferrous salt and nitric acid, add sufficient distilled water to make the solution weigh 100 gm. If the solution is cloudy, filter.

66. *Solution of Ferric Citrate.*

Solution of Tersulphate of Iron.....	52.5 Gm.
Water of Ammonia, 10%.....	44 Gm.
Citric Acid	15 Gm.
Water, a sufficient quantity.	

Dilute the solution of iron tersulphate with 500 mils of water, and the ammonia water with 150 mils of water.

To the diluted ammonia water contained in a suitable vessel (preferably a precipitation jar) add the iron solution with constant stirring, and allow to settle.

If the water of ammonia is deficient in strength, the supernatant liquid will show a yellowish color. If it has color, add ammonia water until the odor of ammonia is apparent after stirring.

When the precipitate has settled, carefully draw off the clear liquid by means of a siphon. Add about 300 mils of water to the precipitate, stir the mixture, and again allow it to subside, and remove the clear liquid again by means of a siphon. Repeat this process about twice more.

Finally, transfer the precipitate to a wetted muslin on a funnel, and when the liquid has passed through, wash the precipitate with distilled water until the filtrate ceases to give more than a slight cloudiness upon the addition of a drop of solution of barium chloride.

Allow the precipitate to drain, transfer to a beaker with a spatula, add the citric acid and heat on a water-bath at a tem-

perature of 60° C., stirring constantly, until solution is effected. Filter the liquid and evaporate on a water-bath until it weighs 50 gm.

67. *Scaled Ferric Citrate.*

Solution of Ferric Citrate, a sufficient quantity.

Evaporate the solution on a water-bath to a syrupy consistence and spread on a clean plate of glass to dry. When dry, remove the scales and preserve in a well-stoppered bottle.

68. *Solution of Dialyzed Iron.*

Solution Ferric Chloride.....	100 Gm.
Aqua Ammonia	35 Gm.

Place the solution ferric chloride in a beaker of suitable size, and add the aqua ammonia very slowly with constant stirring. The precipitate which forms will be re-dissolved, at first readily, but slowly towards the last. Stir until solution is complete.

Place the solution in a dialyzer, and dialyze until the water in the outer vessel, which should be changed frequently, does not give a precipitate with test solution of silver nitrate.

Remove 2 gm. of the liquid from the dialyzer, place on a watch glass, and dry on a water-bath to constant weight.

Determine the weight of the remaining liquid, calculate the amount of solid matter (ferric oxychloride) which it contains, and dilute with distilled water so that the finished product shall contain 10% of ferric oxychloride.

NOTE.—A simple form of dialyzer may be made from a plain beaker by filing the edge of the bottom with a three-cornered file until a small hole has been made, through which the air may enter to keep the inner and outer pressure equal. The dialyzing medium is to be tied over the top.

As a dialyzing medium parchment paper may be used. Any holes in the paper may be sealed by using flexible collodion.

69. *Lead Plaster.*

Lead Oxide	1000 Gm.
Olive Oil	1000 Gm.
Lard	1000 Gm.
Boiling Water, a sufficient quantity.	

Heat the olive oil and lard by a gentle heat until liquefied in a bright copper or other suitable vessel of a capacity of not less than four times the bulk of the ingredients, sift the lead oxide, through a No. 80 sieve, upon the surface of the hot liquid and mix thoroughly. Then gradually add 350 mils of boiling water and boil the mixture, constantly stirring with a wooden spatula, and adding sufficient boiling water from time to time to replace that lost by evaporation, until the mass is homogeneous and a small portion removed and dipped into cold water is found to be pliable and tenacious. Then remove it from the fire and wash several times to remove the glycerin. Finally knead the mass until it is free from water, roll it into cylinders of suitable size and wrap them in paper.

70. *Spirit of Nitrous Ether.*

(Sweet Spirit of Nitre.)

Sodium Nitrite	20 Gm.
Sulphuric Acid	8 Mils.
Monohydrated Sodium Carbonate.....	0.2 Gm.
Potassium Carbonate, perfectly dried..	0.6 Gm.
Alcohol, sufficient.	
Water, sufficient.	

Add the sulphuric acid to 24 mils of water, mix and cool, add 17 mils alcohol diluted with an equal volume of water, and introduce the mixture into a 200 mil flask surrounded by water containing crushed ice.

Dissolve the sodium nitrite in 56 mils of water, filter and pour into a separatory funnel.

Support the separatory funnel over the mouth of the flask containing the acid mixture and allow the sodium nitrite solution to drop slowly into the latter. When all has been added, and the reaction has ceased, allow any crystals which may have formed to subside, decant the mixture of ethyl nitrite and aqueous liquid into the previously cleaned separatory funnel, draw off and discard the aqueous liquid.

Wash the ethyl nitrite in the funnel first with 20 mils of ice water, and then with 15 mils ice water in which the monohydrated sodium carbonate has been dissolved, drawing off the wash water each time.

Carefully separate the ethyl nitrite from any water remaining in the funnel, and transfer it to a clean, dry vial containing the potassium carbonate and agitate to remove the remaining traces of water.

Filter the dried ethyl nitrite through a pledget of cotton into a tared bottle containing 100 mls of alcohol. Ascertain the increased weight of the bottle and contents and add enough alcohol to make the entire product weigh twenty-two times the weight of the ethyl nitrite.

Preserve the product in small, completely filled, dark amber colored vials, in a cool place.

When assayed by the process given in Part Seven, the spirit should contain not less than 4% of ethyl nitrite.

71. *Sulphurous Acid.*

Sulphuric Acid 30 Mils.

Charcoal, in coarse powder..... 10 Gm.

Water, a sufficient quantity.

Introduce the charcoal into a flask having a capacity of about 250 mls, add the acid and mix them well. Provide a wash bottle of about 100 mls capacity, filled to about one-third its height with water, fitted with a stopper having three perforations.

Into a bottle having a capacity of about 750 mls introduce 250 mls of water which has been recently boiled to expel air.

Into still another bottle place about 200 mls of a weak solution of sodium carbonate. (Made by dissolving 10 gm. sodium carbonate in 200 mls of water.)

By means of suitable glass tubing and supports, connect the flask containing the acid and the charcoal with the wash bottle, and that with the bottle containing the water and the latter with the bottle containing the solution of sodium carbonate.

Pass a safety tube through the stopper of the wash bottle, forcing it down until the end nearly reaches the bottom.

The connecting tube entering the water should dip about $2\frac{1}{2}$ cm. below the surface, and that entering the solution of sodium carbonate should be well immersed. (See Cut.)

When the adjustment is complete, apply a moderate heat to the flask containing the acid and charcoal. Continue the heat until the evolution of gas has nearly ceased, keeping the bottle containing distilled water at or below 10° C. by surrounding it with cold water or ice.

Finally, assay the solution of gas by the process given in Part Five, and dilute it with distilled water so that it shall contain 6.4% of sulphurous acid gas.

72. *Pyroxylin.*

Purified Cotton	10 Gm.
Nitric Acid	140 Gm.
Sulphuric Acid	220 Gm.
Water, sufficient.	

Mix the acids gradually in a glass or porcelain vessel, and when the temperature falls to 32° C. add the cotton.

By means of a glass rod mix the cotton thoroughly with the acids, and allow it to macerate until a sample taken out, washed with water and then with alcohol and pressed, is soluble in a mixture of one volume of alcohol and three volumes of ether.

Remove the pyroxylin from the acids and wash thoroughly with cold and then with boiling water until the washings cease to redden blue litmus paper.

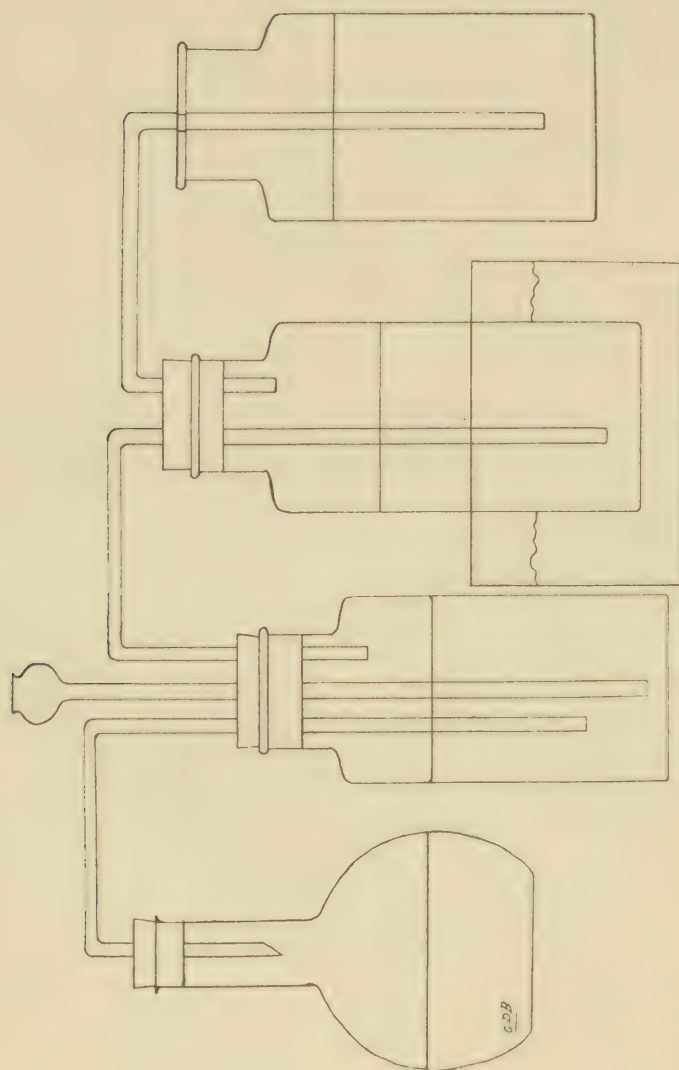
Drain on filter paper, divide it into small pellets and dry on a water-bath at a temperature not above 60° C. Finally, keep the pyroxylin loosely packed in well-closed vessels in a cool, dark place.

CAUTION.—The combustible or even explosive tendencies of this product must be kept in mind.

73. *Phosphoric Acid.*

Phosphorus	16 Gm.
Nitric Acid,	
Distilled Water, of each, a sufficient quantity to make 100 Gm.	

Mix 100 gm. nitric acid with 100 gm. distilled water, in a glass retort having a capacity of about 400 mls.



SULPHUROUS ACID APPARATUS

Having placed the retort on a sand-bath, connect it loosely with a well-cooled receiver and add the phosphorus previously cut into small pieces.

Insert a funnel through the tubulure of the retort and carefully apply heat until the reaction is seen to commence.

Regulate the heat carefully so as to prevent the reaction from becoming too violent and if necessary check it by the addition of a little distilled water through the funnel. From time to time return to the retort the acid liquid which collects in the receiver, until all the phosphorus is dissolved.

Transfer the liquid to a tared porcelain capsule and continue the heat at a temperature not exceeding 190° C. until the excess of nitric acid is driven off and an odorless, syrupy liquid remains.

Place a few drops of the acid in a test tube, with an equal amount of sulphuric acid, and drop in a small crystal of ferrous sulphate. Should a black or brownish color appear around the crystal, nitric acid is present and the liquid must be again heated until the nitric acid is all driven off.

Finally, dilute with distilled water to 100 gm.

74. *Precipitated Sulphur.*

Sublimed Sulphur	50 Gm.
Lime	25 Gm.
Hydrochloric Acid,	
Water, each, a sufficient quantity.	

Slake the lime and make it into a uniform mixture with 250 mls of water. Add the sulphur previously well dried and sifted, mix well, add 500 mls of water, and heat to boiling for one hour, stirring constantly, and replacing the water lost by evaporation.

Cover the vessel and allow to cool, pour off the clear solution, filter the remainder and mix the clear liquids.

Dilute some hydrochloric acid with an equal volume of water and add to the mixed liquids with stirring, until the mixture is *nearly* neutralized, but still retains an alkaline reaction.

Collect the precipitate on a fine muslin strainer and wash with water until the washings are tasteless.

Dry the precipitate with a gentle heat.

75. *Solution of Zinc Chloride.*

Metallic Zinc in small pieces.....	12 Gm.
Hydrochloric Acid	42 Gm.
Precipitated Zinc Carbonate	0.5 Gm.
Nitric Acid	0.5 Mil.
Distilled Water sufficient to make	50 Gm.

Place the zinc in a beaker or evaporating dish, add the hydrochloric acid and allow to stand until the reaction entirely ceases.

Filter through paper into a porcelain dish and add the nitric acid.

Place on a sand-bath and evaporate to dryness, and when a syrupy consistence is reached, stir with a glass rod so as to obtain the salt in a granular condition, and continue the heat until partial fusion takes place.

When cold dissolve in about 20 mls distilled water, add the zinc carbonate, transfer the mixture to a beaker and allow to stand 24 hours, or until the next laboratory period.

Filter through asbestos wool placed in the neck of a funnel, collecting the filtrate in a tared beaker, and add sufficient distilled water through the funnel to make the liquid weigh 50 grams.

Preserve in a glass stoppered bottle.

76. *Solution of Hydrogen Dioxide.*

Barium Dioxide	30 Gm.
Phosphoric Acid	10 Mils.
Distilled Water sufficient to make	100 Mils.

Place the barium dioxide in a 200 mil flask and add 50 mls distilled water, in divided portions, shaking after each addition.

Shake the mixture every few minutes for half an hour.

In another 200 mil flask mix the phosphoric acid with 32 mils distilled water, and remove about 10 mils for subsequent use.

To the dilute acid remaining in the flask add the barium dioxide mixture, in four portions, shaking thoroughly after each addition.

Test the mixture with litmus paper. If the reaction is alkaline, add the reserved dilute acid, a little at a time, shaking after each addition, until the litmus paper turns red.

Set the mixture aside until the precipitate measures about one-third of the volume of the whole.

Pass the clear liquid through a plain filter and collect the filtrate in a graduated cylinder.

Transfer the precipitate to the filter and wash the flask and the precipitate with sufficient distilled water to make the liquid in the cylinder measure 100 mils.

Transfer the liquid to an amber glass bottle, cork loosely, and preserve in a cool place until wanted for assay by the process given in Part Five.

PART FOUR.

PRESCRIPTION PRACTICE.

77.

- ℞ Pulveris rhei gr. XXIV
Magnesiæ gr. XLVIII
Olei anisi m. VI
Alcoholis m. X
M. Tere simul, et in chartulas VI divide.
Sig. One at night.

Place the magnesia in a wedgewood mortar, add the remaining ingredients and triturate until particles of magnesia are no longer visible.

78.

- ℞ Cinchonidiæ sulphatis gr. II
Ammonii carbonatis gr. IV
Misce et dentur tales quartuor in capsulis.
Sig. Sumatur una nocte manequæ.

79.

- ℞ Magnesii carbonatis 1.0 Gm.
Sodii bicarbonatis 14.5 Gm.
Potassii bicarbonatis 8.0 Gm.
Acidi citrici 9.0 Gm.
Acidi tartarici 12.0 Gm.
Sacchari pulveris 5.5 Gm.
Alcoholis, q. s.
Misce. Fiat pulvis effervescens, s. a. Da in ampula.
Sig. Effervescing Powder.

Reduce the solids to powder separately, mix thoroughly and place in a porcelain dish. Add alcohol gradually, while stirring with a wooden spatula or a glass rod, until the powder

coheres in granules. Do not add sufficient alcohol to form a paste. Lastly, dry in a warm place. The operation does not succeed well in a moist atmosphere.

Repeat the above prescription, but granulate according to the dry method, as follows: Place the well mixed powders in an evaporating dish over a water-bath, apply heat and stir with a glass rod or wooden spatula until converted to granules of fairly uniform size. Spread on a piece of paper until cool.

The powder should be preserved in a wide mouth bottle, well corked and the cork covered with paraffin or tin foil.

80.

R	Iodi	0.4
	Potassi iodidi	0.1
	Aquæ	0.2
	Adepis benzoinati	9.3
	Misce. Fiat unguentum.	

Dissolve the potassium iodide in the water by triturating in a mortar, add the iodine and rub until particles are no longer visible. Incorporate the benzoinated lard gradually, and remove from the mortar with a non-metallic spatula.

81.

R	Zinci oxidi	1.0
	Camphoræ	0.15
	Unguenti	20.0
M.	Fiat unguentum.	
Sig.	Eye Salve.	

Melt $\frac{1}{2}$ of the ointment, and dissolve the camphor in the warm fat. Incorporate the zinc oxide by triturating with a small portion of the ointment till perfectly smooth, then add the melted camphor and fat and mix all together.

82.

R	Camphoræ	
	Chloralis a. a.	4.0
	Petrolati	30.0
M.	Ft. ungt.	
Sig.	Ad partem dolentum applicetur.	

Triturate the camphor and chloral in a mortar until liquefied and incorporate the petrolatum.

83.

℞ Naphtoli 2.0
 Petrolati 20.0
 Aetheris, q. s.
 Misce. Fiat unguentum.
 Sig. Ut dictum.

Dissolve the naphthol in 3 mls of ether and mix with 5 gm. petrolatum. Warm on a water-bath till the ether is expelled, and incorporate the remainder of the petrolatum.

84.

℞ Cetacei 6.25
 Cera albæ 6.00
 Olei amygdalæ express 30.00
 Aquæ rosæ fort 9.50
 Sodii boratis, pulv. 0.25
 M. S. Cold Cream. Apply to chapped or inflamed parts.

Reduce the spermaceti and white wax to fine shavings with a knife, melt in a capsule, add the oil of almond, and transfer to a warm mortar. Dissolve the borax in the rose water, and add all at once, without stirring. Then stir rapidly and continuously until soft and creamy.

NOTE.—The mortar may be warmed by filling with hot water, then wiping dry.

85.

℞ Pulv. glycyrrhizæ comp. 3 iv
 Pulpæ pruni, q. s.
 M. Ft. trochisci No. XX.
 Sig. Compound Licorice Lozenges.

About two *small* prunes will be required. Stew about two minutes, and remove the skin. *Caution:* Add only sufficient of the prune pulp to make a workable mass.

86.

R Acidi arsenosi gr. j
 Sacchari lactis, q. s.
 Misce. Fiant tabellæ triturationes No. L.
 Sig. Arsenous Acid Tablets $\frac{1}{60}$ grain. POISON.

Mix thoroughly the arsenous acid and milk sugar, moisten with alcohol, and fill the holes of the triturate machine.

First ascertain how much milk sugar will be required to fill the holes when used alone, then take one grain less. If water be used as the moistening agent, the mass will be more compact, and a larger amount of milk sugar will be required.

87.

R Pulv. glycyrrhizæ comp. gr. 60

Granulate and make 12 tablets by compression.

88.

R Gossypii purificati 10.00
 Liq. ferri chloridi 8.00
 Glycerini 1.60
 Aquæ 11.25
 Fiat gossypium stypticum. Detur in scatula.
 Sig. Styptic Cotton. N. F.

Place the cotton in a compact roll in a mortar, pour on the mixed liquids. Press repeatedly with the pestle, cover the mortar and allow to stand for several hours. Lastly, unroll the cotton, and dry in the dark. Preserve in a tightly closed vessel.

89.

R Camphoræ 2.5
 Saponis pulv. 0.39
 Olei ricini, q. s.
 Fiant pilulæ No. XII.
 Sig. 3 gr. Camphor Pills.

Triturate the camphor in a mortar with a little alcohol or ether, until the liquid has evaporated and a dry powder re-

mains. Add the soap and mix thoroughly, and then add castor oil, drop by drop, with constant kneading, until a workable mass is produced.

90.

℞ Ferri citratis 2.0
 Ext. glycyrrhizæ, pulv.
 Syr. glucosi, a a q. s.
 Fiant pilulæ No. X.
 Sig. Cap. unam post cibo.

91.

℞ Cinchonidiæ sulphatis 1.3
 Syr. glucosi, q. s.
 Ft. Pil. No. XX. Consperge amylo.
 Sig. More dictu.

92.

℞ Pot. permanganatis gr. 8
 Kaolini gr. 30
 Petrolati, q. s.
 M. Ft. pil. No. X.
 Sprinkle with kaolin.
 Sig. Permanganate Pills.

Powder and mix the solids, add sufficient petrolatum to make a workable mass. Mass and roll quickly.

93.

℞ Creosoti 2 Mils.
 Saponis pulv., q. s.
 Ft. pil. No. XX
 Sig. Creosote Pills.

Use just sufficient of powdered soap to make a workable mass.

94.

℞ Chloralis
 Camphoræ a a 1.00
 Farinæ tritici 3.50
 Syr. glucosi, q. s.
 Ft. pil. No. X.
 Sig. Chloral and Camphor Pills.

Triturate the camphor and chloral in a mortar until liquefied and add sufficient wheat flour to make a mass.

95.

- ℞ Strychninæ sulphatis 0.032
 Acidi arsenosi 0.065
 Ferri sulphatis exsiccati 0.650
 Ext. glyc. pulv. 1.500
 Syr. glucosi, q. s.
 M. Ft. massa secundem artem et in pil. XX dividenda.

CAUTION.—Be sure that the first two ingredients are thoroughly powdered and evenly distributed through the mass.

96.

- ℞ Potassii iodidi gr. xv
 Amyli gr. xl
 Syrupi glucosi, q. s.
 Fiant pilulæ No. XV.
 Sig. Capiat unam post cibo.

Powder the salt finely, mix with the starch and mass with the glucose.

97.

- ℞ Potassii iodidi gr. xv
 Olei theobromatis gr. xv
 Lanolini, q. s.
 M. Ft. pil. No. X.
 Sig. One pill as directed.

Powder the salt, incorporate the cacao butter and add lanolin q. s. to mass.

98.

- ℞ Olei tiglii gtt. vi
 Farinæ tritici gr. xx
 Saponis gr. ii
 Syr. glucosi, q. s.
 Ft. pil. No. vj.
 Sig. Croton Oil Pills.

99.

R	Plumbi acetatis	1.20.
	Opil pulv.	0.40.
	Olei theobromatis, q. s.	
	Fiant suppositoria, pro recto, No. VI.	
	Sig. Ut dictum.	

Mix the powdered ingredients and mould by the Allen Suppositor or other suppository machine.

100.

R	Chloralis	2.00
	Cetacei	2.50
	Olei theobromatis, q. s.	
	Fiant suppositoria pro recto, No. VI secundem artem.	
	Sig. Insert one at night.	

Melt the spermaceti and cacao butter on a water-bath, add the chloral, and pour into well chilled moulds.

101.

R	Acidi tannici	0.300
	Acidi carbolici	0.200
	Olei theobromatis	6.000
	Fiant suppositoria, No. VI.	

Make by massing on a tile, with a horn or vulcanite spatula, and shaping into suppositories. Do not use a metallic spatula.

102.

R	Gelatini	2.5
	Glycerini	10.0
	Aquæ destillatæ, q. s.	
M.	Ft. suppositoria pro recto, no. vj.	

Place the gelatin in a weighed evaporating dish, cover with distilled water and let stand two minutes. Pour off the water, cover, and let stand until the gelatin is perfectly soft. Then add the glycerin and heat on a water-bath till reduced to the weight of 13 gm. Pour into cold and well oiled moulds. Preserve in a morphine vial, tightly corked.

103.

℞ Glycerini	15	Gm.
Sodii carbonatis monohydratis	0.25	Gm.
Acidi stearici	1	Gm.
Aquæ	2.5	Mils.

Misce. Fiat suppositoria, No. V.

Sig. Use as directed.

Dissolve the sodium carbonate in the water and add it to the glycerin contained in a dish on a water-bath. Add the stearic acid, continue the heat, with stirring, until carbon dioxide ceases to be evolved and the mixture is clear. Pour the mass into perfectly clean, well chilled moulds, and allow to stand until completely cold. Dispense in tightly closed glass vessels.

104.

℞ Olei jecoris aselli	32	Mils.
Olei gaultheriæ	0.20	Mil.
	(about 4 drops.)	
Acaciæ, pulv.		
Aquæ, a a, q. s. ad.....	90	Mils.

Fiat emulsion.

Sig. Emulsion of Cod Liver Oil.

Place 8 gm. powdered acacia in a mortar, add the oil and mix thoroughly; then add 16 mils of water, all at once, and stir lightly and quickly until the emulsion is formed. Then dilute with the remainder of the water added gradually, with constant stirring. Transfer to a bottle, add the oil of gaultheria and shake.

105.

℞ Olei ricini	f 3 j
Acaciæ pulv.	3 iv
Olei amygdalæ amaræ	gtt. ii
Olei caryophylli	gtt. i
Saccharini	gr. i
Aquæ, q. s.	f 3 iv

M. Ft. emulsio secundum artem.

Sig. Pro alvo astricta.

Proceed as with the preceding emulsion, using twice as much water as acacia to make the primary emulsion, before diluting with the remainder.

106.

R	Saponis, pulv.	4 Gm.
	Benzini	30 Mils.
	Aquæ ammoniæ	30 Mils.
	Aquæ, q. s. ad	120 Mils.
	Fiat emulsum secundum artem.	
	Sig. Benzine Jelly.	

Dissolve the soap in a capsule in 40 mils water with the aid of heat. Strain, transfer to a 120 mil bottle, and gradually add the benzine, with constant agitation. When emulsified, gradually add the ammonia water, with constant shaking, then water q. s. to make 120 mils.

NOTE.—Use “Curd Soap,” or soap made from animal fat or coconut oil.

107.

R	Olei terebinthinæ purificati	15 Mils.
	Olei amygdalæ expressi	5 Mils.
	Syrupi	25 Mils.
	Acaciæ, pulv.	15 Gm.
	Aquæ, q. s. ad	100 Mils.
	M. Ft. emulsum.	
	Sig. Emulsion of Turpentine	

Introduce the acacia into a clean, dry bottle. Add the oils of turpentine and almond, and shake thoroughly. Add 30 mils of water and again shake vigorously until the emulsification is completed. Add the syrup in divided portions, shaking vigorously after each addition, and then the remainder of the water in portions, with shaking.

108.

R	Asafoetidæ	℥ iss
	Aquæ, q. s. ad	f ⅔ iv
	Fiat emulsum.	
	Sig. Cochleare magnum ter in die.	

Use selected tears of asafœtida, not the powdered drug. Place the tears in a mortar, add water very gradually, with constant trituration, until a smooth mixture results.

Strain through muslin into a graduated measure, and wash the mortar and strainer with sufficient water to make 4 fluidounces.

In cold weather use a warm mortar and warm water.

109.

℞ Liq. ferri chloridi f ʒ j
 Mucilag. acaciæ f ʒ iv
 Aquæ, q. s. ad. f ʒ iij
 Misce. secundum artem.
 Sig. Coch. parv. ter in die.

If the mucilage and solution of ferric chloride be mixed directly together, an intractable gelatinous mass will be produced.

Therefore, proceed as follows: Place the ferric chloride solution in a bottle and dilute with half the water. Dilute the mucilage with the remaining water, and add in small portions to the liquid in the bottle, shaking after each addition.

110.

℞ Potassii chloratis pulv. gr. x
 Acidi hydrochlor. f ʒ iss
 Aquæ, q. s. ad. f ʒ iij
 Fiat solutio. Sig. Ut dictum.

Place the powder in a two-ounce bottle, add the acid, insert the cork, and allow to stand until the mixture becomes a deep yellow, and fill up with cold water.

The purpose of this manipulation is to furnish a strong solution of free chlorine, and certain oxides of chlorine.

If the salt is dissolved in the water before the acid is added, the gas will not be set free, unless after a very long time.

PART FIVE.

VOLUMETRIC ANALYSIS.

111. *Preparation of Normal Oxalic Acid Solution.*

Weigh out accurately 63 gm. pure crystallized oxalic acid and dissolve it with the aid of a gentle heat in about 800 mls of distilled water contained in a 1000 mil graduated flask. When all is dissolved, fill the flask to the 1000 mil mark, agitate and cool to room temperature. Fill again to the mark and agitate.

Each mil of this solution contains .063 gm. $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$ and will exactly neutralize 1 mil of any *normal* alkaline solution.

112. *Preparation of Normal Sodium Hydroxide Solution.*

1 Liter = 40 gm. NaOH.

Sodium hydroxide is of variable strength and it is therefore necessary in making the normal solution to take a greater quantity than 40 gm. to the Liter and dilute the solution after **ascertaining its strength.**

Weigh out 50 gm. NaOH and dissolve it in sufficient distilled water, which has been previously boiled to expel carbon dioxide, to make one Liter. After thoroughly mixing remove 10 mls to a small beaker and add a few drops phenolphthalein solution. From a burette run in normal oxalic acid until the pink color is just discharged. Repeat the titration until several concordant results are obtained. From the number of mls of the acid solution used the strength of the NaOH solution may be determined and the amount of dilution necessary to make it normal. If, for example, 11.1 mls of the normal acid solution were required to neutralize 10 mls of the sodium hydroxide solution, enough distilled water must be added to make

each 10 mls of the hydroxide solution measure 11.1 mls or to 900 mls of the sodium hydroxide solution 99 mls of water must be added to make it normal. If the determination was correct, 10 mls of this solution will now exactly neutralize 10 mls of the normal acid.

113. Preparation of Normal Hydrochloric Acid Solution.

1 Liter = 36.47 gm. HCl.

Mix 120 mls hydrochloric acid (specific gravity 1.155) with sufficient distilled water to make one Liter. After thorough agitation transfer 10 mls, by means of a pipette, to a beaker, add a few drops of phenolphthalein solution and from a burette add normal sodium hydroxide until a permanent pink color is produced after stirring.

Calculate the strength of the acid solution and dilute with water so that 10 mls just equals 10 mls of the normal NaOH.

114. Preparation of Normal Sulphuric Acid Solution.

1 Liter = 49.045 gm. H₂SO₄.

Sulphuric acid being a dibasic acid only one-half of the molecular weight in grammes is taken for one Liter of normal solution.

Make a dilute sulphuric acid by mixing 30 mls pure sulphuric acid (sp. gr. 1.83) with water sufficient to make about one Liter, and after cooling to the room temperature, transfer 10 mls to a beaker and titrate with normal NaOH using phenolphthalein as indicator. After ascertaining its strength dilute the acid solution and make it normal.

ALKALIMETRY.

115. Determination of the Quantity of Sodium Hydroxide Contained in a Solution of Unknown Strength.

Rinse the burette with a few mls of the normal oxalic acid solution and fill it with the solution. With a pipette meas-

ure 5 mls of the NaOH solution and run it into a beaker, add 20 mls distilled water and 2 or 3 drops of phenolphthalein solution.

Make a note of the reading of the burette and slowly add the normal oxalic acid solution to the contents of the beaker, with constant stirring, until the red color is just discharged. Again note the reading of the burette and record the number of mls of the acid solution used.

The titration is to be repeated until at least two concordant results are obtained.

116. *Calculation of Analysis.*

Supposing that it required 7.2 mls normal oxalic acid to neutralize the 5 mls NaOH solution, then, as one mil normal oxalic acid will exactly neutralize one mil normal NaOH, if we know how much NaOH is contained in one mil of normal NaOH it is easy to calculate the quantity of NaOH contained in the 5 mls of the solution tested.

Normal solutions contain the hydrogen equivalent of a substance expressed in grams in one Liter.

The hydrogen equivalent of NaOH being 40, normal NaOH would contain 40 gm. NaOH to the Liter or 40 milligrams to the mil, then

1 mil normal oxalic acid = 1 mil normal NaOH.

7.2 mls normal oxalic acid = 7.2 mls normal NaOH.

1 mil normal NaOH = .040 gm. NaOH.

7.2 mls normal NaOH = $7.2 \times .040$ gm. NaOH (or .288 gm.)

and 5 mls NaOH solution contains .288 gm. NaOH.

117. *Use of Methyl Orange as an Indicator.*

Methyl orange is yellow in alkaline and red in acid solutions. While the change from yellow to red is gradual, the exact point of neutralization may be readily determined with a little practice, the liquid changing from a bright golden yellow to a flesh color. The smallest possible quantity of the indicator should be used to get the best results; large quantities color

the liquid orange instead of bright golden yellow. In order to familiarize himself with these color changes, the student should repeat the following blank experiment until he can distinguish with certainty the exact point of neutralization.

Add to 50 mls distilled water one drop of methyl orange solution and one or two drops of decinormal NaOH and note the bright golden yellow color. Now add, one drop at a time, decinormal H_2SO_4 , noting the changes in color. At the second or third drop of acid there should be a faint, yet perfectly distinct change of color, which indicates a change from alkaline to acid reaction. The further addition of the acid produces a cherry red.

118. *Determining the Strength of Sodium Carbonate Solution.*

Fill the burette with normal HCl after rinsing with a few mls of the same.

Measure 5 mls Na_2CO_3 solution in a beaker, add 20 mls distilled water and one drop of methyl orange solution as indicator. Run in the normal HCl until the bright yellow color just changes to flesh color.

Calculate the quantity of Na_2CO_3 contained in the 5 mls of solution.

119. *Method of Calculation.*

Supposing 9.8 mls normal HCl were required to neutralize the 5 mls Na_2CO_3 solution, then

$$\begin{aligned} 1 \text{ mil normal HCl} &= 1 \text{ mil normal Na}_2\text{CO}_3. \\ 9.8 \text{ mls normal HCl} &= 9.8 \text{ mls normal Na}_2\text{CO}_3. \end{aligned}$$

The reaction taking place being

$$\begin{array}{r} \text{Na}_2\text{CO}_3 + 2\text{HCl} = 2\text{NaCl} + \text{CO}_2 + \text{H}_2\text{O}. \\ 2) \quad 106 \qquad 72.94 \\ \hline \qquad 53 \qquad 36.47 \end{array}$$

As it requires 72.94 grams of hydrochloric acid to neutralize 106 grams of sodium carbonate, 36.47 grams of hydro-

chloric acid, the amount contained in one Liter of normal HCl, would neutralize 53 grams of sodium carbonate, and as normal solutions are so made that they neutralize each other volume for volume, one Liter of normal sodium carbonate solution would contain 53 grams and

1 mil normal $\text{Na}_2\text{CO}_3 = .053 \text{ gm. Na}_2\text{CO}_3$.

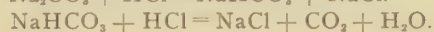
9.8 mils normal $\text{Na}_2\text{CO}_3 = .053 \times 9.8 \text{ gm. Na}_2\text{CO}_3$ (or .5194 gm.).

The 5 mils of sodium carbonate solution therefore contained .5194 gm. of sodium carbonate.

120. Carbonates with Phenolphthalein as Indicator.

Repeat the last titration, using phenolphthalein instead of methyl orange as indicator, and note the difference in results.

This is due to the fact that phenolphthalein does not show an alkaline reaction with NaHCO_3 , while methyl orange does. The reactions taking place being:



The solution becomes colorless when the neutralization is just half completed.

121. Estimation of Ammonia Water.

Fill a burette with the ammonia water to be tested, after rinsing the burette with a few mils of the same.

Carefully run 10 mils into a tared and glass stoppered weighing bottle and weigh accurately. By moving the decimal point one place to the left the specific gravity is obtained.

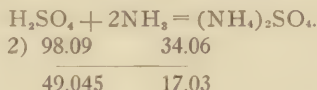
Into a beaker containing 20 mils distilled water and five drops of rosolic acid T. S., measure accurately from the burette 3 mils of the ammonia water and titrate with normal H_2SO_4 until the color changes to yellow.

122. *Calculation of Analysis.*

Suppose the 10 mls of ammonia water weighed 9.6 gm., the specific gravity would be .960. The 3 mls used would then weigh $3 \times .960$, or 2.88 gm. If it required 16.9 mls normal sulphuric acid to neutralize this, then

$$\begin{aligned} 1 \text{ mil normal H}_2\text{SO}_4 &= 1 \text{ mil normal ammonia.} \\ 16.9 \text{ mls normal H}_2\text{SO}_4 &= 16.9 \text{ mls normal ammonia.} \end{aligned}$$

The reaction taking place being



A Liter of normal ammonia would therefore contain 17.03 grams NH_3 and one mil, 17.03 milligrams.

$$\begin{aligned} 1 \text{ mil normal ammonia} &= .01703 \text{ gm. NH}_3. \\ 16.9 \text{ mls normal ammonia} &= .2878 \text{ gm. NH}_3 \ (16.9 \times .01703), \\ \text{and } .2878 \div 2.88 &= .0999 \text{ or } 9.99\%. \end{aligned}$$

123. *Estimation of Potassium Bitartrate.*

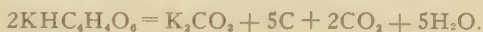
Organic salts of the alkalies and alkaline earths yield, when ignited, carbonates and oxides and may be readily estimated by ignition and estimation of the carbonate or oxide produced.

The salt to be examined should be thoroughly dried before weighing. If it contains no water of crystallization it should be placed in an air-bath at 100°C . for several hours, otherwise powder finely and place in a desiccator over calcium chloride for 24 hours.

Heat about 2 gm. of the salt to be assayed, accurately weighed, in a platinum or porcelain crucible, at first very gently, then gradually raise the temperature until the salt is thoroughly carbonized. (Do not use platinum crucibles for lithium salts.) The final temperature must not exceed a dull red heat and the flame of the burner must not come in contact with the carbonized mass. After allowing the carbonized mass

to cool, disintegrate it with the aid of a stout glass rod and transfer the mass and crucible to a beaker. Add 50 mls of distilled water and 50 mls of half-normal sulphuric acid V.S., cover the beaker with a watch glass and boil the contents for thirty minutes. Then filter the solution and wash the residue with hot distilled water until the washings cease to redden blue litmus paper. Now determine the residual acid in the cooled filtrate by titration with half-normal potassium hydroxide V.S., using methyl orange T.S. as indicator. The difference between the number of mls of half-normal sulphuric acid V. S. added and the number of mls of half-normal potassium hydroxide V. S. used, multiplied by the respective equivalent of the salt represents the amount of salt present in the amount taken.

Upon ignition two molecules of potassium bitartrate yield one molecule of potassium carbonate as follows:



ACIDIMETRY.

124. *Determining the Strength of Dilute Sulphuric Acid.*

Fill the burette with normal NaOH after rinsing with a few mls of the same.

Measure 5 mls of the acid to be tested into a beaker, add 20 mls water and one drop of methyl orange solution. Add the normal NaOH from the burette until a yellow color is produced.

Calculate the quantity of H_2SO_4 in the 5 mls of acid.

Repeat the test, using phenolphthalein as indicator.

125. *Estimation of Acetic Acid.*

Weigh into a beaker from 3 to 5 grams of acetic acid and after diluting with 20 mls of water and adding two or three drops of phenolphthalein solution, titrate with normal NaOH until a pink color remains after stirring.

Calculate the percentage of absolute acetic acid.

ANALYSIS BY PRECIPITATION.

126. Valuation of Lead Subacetate Solution.

Measure with a pipette 10 mls lead subacetate solution into a tared beaker and weigh accurately, add 30 mls hot distilled water, which has just been well boiled, and three drops methyl orange solution. Add normal H_2SO_4 until precipitation ceases and the liquid has assumed a pink color.

From the number of mls of normal H_2SO_4 used calculate the amount of lead subacetate in the 10 mls of solution and then the percentage strength.

Verify the result by adding a slight excess of the normal H_2SO_4 to the contents of the beaker and collecting the lead sulphate on a double plain filter, the filter papers having been dried and counterpoised. Wash the precipitate with dilute alcohol, dry, first in the air, then in a drying oven, and weigh.

From the weight of the lead sulphate obtained, calculate the percentage of lead subacetate in the solution.

As the formula for lead subacetate use $\text{Pb}(\text{PbO})(\text{C}_2\text{H}_3\text{O}_2)_2$.

127. Preparation of Decinormal Silver Nitrate.

1 Liter = 16.989 gm. AgNO_3 .

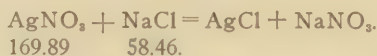
1 mil = .016989 gm. AgNO_3 .

Dissolve 16.989 gm. of pure silver nitrate in sufficient distilled water to make the volume of the solution one Liter.

128. Determination of Sodium Chloride.

Measure into a beaker, with a pipette, 10 mls of solution of sodium chloride, add 20 mls distilled water and two or three drops of potassium chromate solution. After rinsing a glass stoppered burette with a few mls of the decinormal silver nitrate, fill the burette with the same and run it into the sodium chloride mixture until a faint pink color remains after stirring.

One molecule of the silver nitrate precipitates one molecule of sodium chloride.



1 mil decinormal AgNO_3 is therefore equivalent to .005846 NaCl .

OXIDIMETRY.

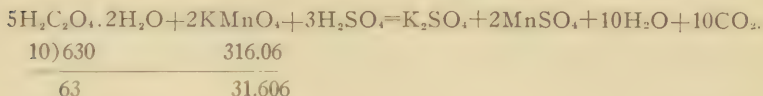
Evaluation of Potassium Permanganate Solution and Preparation of Decinormal Potassium Permanganate Solution.

Dissolve about 3.5 grams pure crystallized potassium permanganate in one Liter of boiling distilled water, filter through a pledget of glass wool, rejecting the first few mls of filtrate, and cool to room temperature.

With a pipette measure with great accuracy 10 mls decinormal oxalic acid into a beaker, add 10 mls distilled water and 2 mls concentrated H_2SO_4 .

After rinsing a glass stoppered burette with the solution of potassium permanganate fill it with the same and add slowly to the contents of the beaker until a permanent pink color is produced. The quantity of permanganate solution used is equal to one mil of normal potassium permanganate solution.

Two molecules of potassium permanganate give off five atoms of oxygen in the presence of an oxidizable body in acid solution. In the present case the following reaction takes place:



Assuming that it took 9 mls permanganate solution to oxidize one mil of normal oxalic acid, then

9 mls KMnO_4 solution = 1 mil normal oxalic acid.
 1 mil normal KMnO_4 = 1 mil normal oxalic acid.
 therefore 9 mls KMnO_4 solution = 1 mil normal KMnO_4 .
 and 1 mil KMnO_4 solution = $\frac{1}{9}$ mil normal KMnO_4 , or .1111 mil.

Usually this factor is taken as the value of the permanganate solution and the label made to read

1 mil = .1111 mil normal or 1.111 mls decinormal Potassium Permanganate.

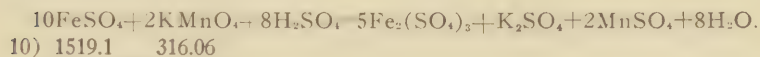
The solution may, however, be readily made decinormal by diluting 1000 mls to measure 1111 mls.

129. Estimation of Ferrous Sulphate.

Place 2 mls ferrous sulphate solution in a beaker with 20 mls distilled water and 2 mls concentrated H_2SO_4 and titrate with the above permanganate solution until a permanent pink color is produced.

130. Calculation of Analysis.

Supposing it required 12 mls permanganate solution to oxidize the ferrous sulphate in the 2 mls of solution, then, as each mil of the permanganate solution equals .1111 mil normal, $12 \times .1111$ or 1.3332 mls of normal KMnO_4 were required. The following reaction taking place, and normal KMnO_4 containing 31.606 grams in a Liter, it follows that normal ferrous sulphate would contain 151.91 grams per Liter or 151.91 milligrams in one mil.



10) 1519.1 316.06

151.91 31.606

1 mil normal $\text{KMnO}_4 = 1$ mil normal FeSO_4 .

1.3332 mls normal $\text{KMnO}_4 = 1.3332$ mls normal FeSO_4 .

1 mil normal $\text{FeSO}_4 = .15191$ gm. FeSO_4

1.3332 mls normal $\text{FeSO}_4 = 1.3332 \times .15191$ or .20252.

The 2 mls of solution therefore contained .20252 gm. FeSO_4 .

131. Determination of Ferric Iron after Reduction to Ferrous Iron.

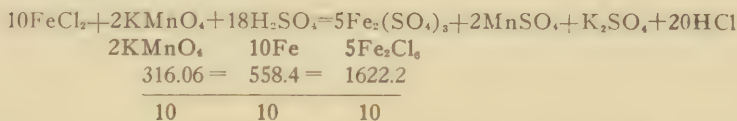
Dissolve about .4 gm. (accurately weighed) ferric chloride in 250 mls water containing 25 mls of sulphuric acid contained in a 500 mil flask provided with a Bunsen valve stop-

per. Add exactly 10 gm. of zinc and place upon a water-bath until the zinc has all dissolved and a drop of the fluid fails to give a red color with potassium sulphocyanide solution. To the cooled solution add decinormal KMnO_4 from a burette until a faint permanent pink color results.

As almost all metallic zinc contains iron it is necessary to make a blank experiment in which 10 gm. of the same zinc are dissolved in 250 mls distilled water and 25 mls H_2SO_4 contained in a 500 mil flask provided with a Bunsen valve stopper, and after cooling run in permanganate solution until a permanent rose tint results.

132. Calculation of Analysis.

Supposing it required 29.2 mls decinormal KMnO_4 to oxidize the iron solution and 7.2 mls for the zinc solution, then $29.2 - 7.2 = 22$, the number of mls required to oxidize the iron from the ferric chloride.



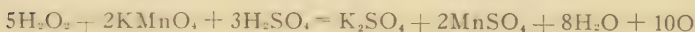
If 1 mil decinormal KMnO_4 is equivalent to .005584 gm. of metallic iron, which in turn is equivalent to .016222 gm. of ferric chloride, then 22 mls would be equivalent to $22 \times .016222$ or .356884 gm. of ferric chloride. The sample weighing .4 gm. contains, therefore, .356884 gm. of true ferric chloride or 89.2%.

133. Valuation of Hydrogen Dioxide Solution.

Place 2 mls of the hydrogen dioxide solution in a beaker with 10 mls water and 10 mls dilute sulphuric acid and titrate with decinormal KMnO_4 until a faint pink color remains after stirring.

Calculate the percentage strength of the solution.

Reaction:

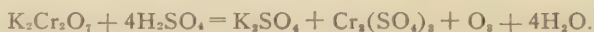


134. *Preparation of Decinormal Potassium Dichromate.*

$$1 \text{ Liter} = 4.903 \text{ gm. } K_2Cr_2O_7.$$

Dissolve 4.903 gm. of pure potassium dichromate crystals in sufficient distilled water to make one Liter.

In the presence of acids potassium dichromate acts as an oxidizing agent and breaks up as follows:



Three atoms of oxygen being the equivalent of six atoms of hydrogen, one-sixth of the molecular weight in grams of $K_2Cr_2O_7$ is taken to make one Liter of the normal solution and one-sixtieth the molecular weight in grams for the decinormal.

135. *Estimation of Ferrous Sulphate by Decinormal $K_2Cr_2O_7$.*

Place 2 mls ferrous sulphate solution in a beaker with 20 mls water and 2 mls concentrated sulphuric acid and add from a burette decinormal $K_2Cr_2O_7$ until a drop of the liquid no longer produces a blue color on filter paper saturated with a freshly prepared solution of potassium ferricyanide.

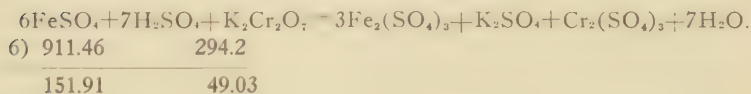
136. *Calculation of Analysis.*

Supposing it required 11 mls decinormal potassium dichromate to oxidize the ferrous sulphate in 2 mls of the solution, then

$$1 \text{ mil decinormal } K_2Cr_2O_7 = 1 \text{ mil decinormal } FeSO_4.$$

$$11 \text{ mls decinormal } K_2Cr_2O_7 = 11 \text{ mls decinormal } FeSO_4.$$

As the following reaction takes place:



then

$$1 \text{ mil decinormal } FeSO_4 = .015191 \text{ gm. } FeSO_4.$$

$$11 \text{ mls decinormal } FeSO_4 = .167101 \text{ gm. } FeSO_4.$$

The 2 mls of solution therefore contain .167101 gm. anhydrous ferrous sulphate.

IODIMETRY.

137. *Preparation of Decinormal Iodine.*

1 Liter = 12.692 gm. Iodine.

Dissolve 12.692 gm. of *chemically pure* iodine in a solution of 18 gm. of potassium iodide (free from iodate) in 300 mls of distilled water. When all is dissolved dilute the solution to make one Liter.

138. *Preparation of Decinormal Sodium Thiosulphate.*1 Liter = 24.822 gm. $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$.

Dissolve 30 gm. of pure crystallized sodium thiosulphate in sufficient distilled water to make one Liter and standardize the solution as follows:

To 10 mls of the sodium thiosulphate solution containing 2 drops of starch solution add from a burette decinormal iodine until the addition of one drop produces a blue color which remains after stirring.

Dilute the thiosulphate solution so that 10 mls will be just equivalent to 10 mls of the decinormal iodine. If it took 13.1 mls to produce the blue color, then every 100 mls must be made to measure 131 mls.

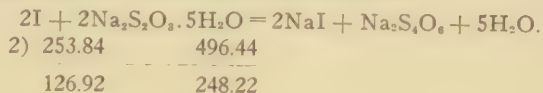
139. *Valuation of Tincture of Iodine.*

Mix 3 mls of tincture of iodine with 15 mls of water and titrate with decinormal sodium thiosulphate. When the yellow color due to the free iodine has almost disappeared add a few drops of starch solution and continue adding the thiosulphate solution slowly until the blue color just disappears on stirring.

Calculate the percentage of iodine in the tincture.

140. *Method of Calculation.*

Iodine reacts with sodium thiosulphate to produce sodium iodide and sodium tetrathionate as follows:



Supposing it took 16.1 mls of decinormal sodium thiosulphate to combine with the iodine in the 3 mls of tincture, then

$$\begin{aligned} 1 \text{ mil decinormal thiosulphate} &= 1 \text{ mil decinormal iodine.} \\ 16.1 \text{ mls decinormal thiosulphate} &= 16.1 \text{ mls decinormal iodine.} \\ 1 \text{ mil decinormal iodine} &= .012692 \text{ iodine.} \\ 16.1 \text{ mls decinormal iodine} &= 16.1 \times .012692 \text{ or } .20434 \text{ gm. iodine.} \end{aligned}$$

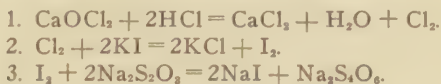
The 3 mls of tincture contain .20434 gm. iodine or 6.81+%, the specific gravity of the tincture not being taken into consideration in this case.

141. *Estimation of Chlorine in Chlorinated Lime.*

Triturate 0.3 gm. chlorinated lime with 30 mls of water, transfer to a flask together with the washings, add 5 mls potassium iodide solution and 3 mls dilute hydrochloric acid and agitate thoroughly. Titrate with decinormal sodium thiosulphate, adding toward the end of the reaction a few drops of starch solution, until the blue color is just discharged.

142. *Method of Calculation.*

The following are the reactions taking place:



Each atom of chlorine liberated by the hydrochloric acid liberates in turn one atom of iodine, therefore 126.92 gm. iodine would represent 35.46 gm. of chlorine or $.012692 = .003546 \text{ Cl}$.

Supposing we used 30.2 mls decinormal thiosulphate, then

$$\begin{aligned} 1 \text{ mil decinormal thiosulphate} &= 1 \text{ mil decinormal iodine.} \\ 1 \text{ mil decinormal iodine} &= .012692 \text{ gm. iodine.} \\ \text{and } .012692 \text{ gm. iodine} &= .003546 \text{ gm. chlorine.} \\ \text{therefore 1 mil decinormal thiosulphate} &= .003546 \text{ gm. chlorine.} \\ \text{and } 30.2 \text{ mls decinormal thiosulphate} &= 30.2 \times .003546 \text{ or } .10708 \\ &\text{gm. chlorine.} \end{aligned}$$

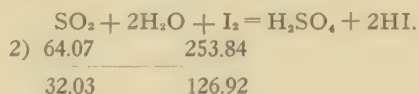
The 0.3 gm. of chlorinated lime contained .10708 gm. of chlorine or 35.69%.

143. Valuation of Sulphurous Acid.

Weigh carefully in a stoppered weighing bottle about 2 mls of sulphurous acid and add 50 mls decinormal iodine solution. Allow to stand for five minutes and add slowly from a burette decinormal sodium thiosulphate solution until the mixture is just decolorized.

144. Method of Calculation.

The sulphurous acid is oxidized to sulphuric acid by the iodine as shown by the following equation:



Supposing 2 gm. of sulphurous acid to have been used, the 50 mls decinormal iodine added contains more iodine than is required to oxidize the SO_2 present, the excess being determined by titration with decinormal thiosulphate. Supposing that it required 12.6 mls decinormal thiosulphate to discharge the color, then

1 mil decinormal thiosulphate = 1 mil decinormal iodine
 12.6 mls decinormal thiosulphate = 12.6 mls decinormal iodine
 50 mls decinormal iodine used, minus 12.6 mls which were decolorized by the thiosulphate, leaves 37.4 mls required to oxidize the SO_2 in 2 gm. sulphurous acid.

1 mil decinormal iodine = 1 mil decinormal sulphur dioxide.
 37.4 mls decinormal iodine = 37.4 mls decinormal sulphur dioxide.
 1 mil decinormal sulphur dioxide = .003203 gm. sulphur dioxide.
 37.4 mls decinormal sulphur dioxide = $37.4 \times .003203$ or .11979 gm. sulphur dioxide.

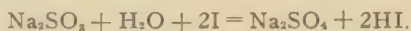
Therefore, 2 gm. of the sulphurous acid contain .11979 gm. SO_2 or 5.89%.

145. Estimation of Sulphites

To 50 mls decinormal iodine contained in a 100 mil glass stoppered flask add about 0.5 gm. crystallized (or 0.25 gm. dried) sodium sulphite, accurately weighed, and allow to stand

for one hour with occasional agitation. Titrate with decinormal thiosulphate until the color is just discharged.

Calculate the strength of the sodium sulphite, the reaction being as follows:

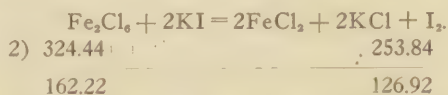


146. Iodometric Estimation of Ferric Iron.

Weigh carefully about 0.3 gm. crystallized ferric chloride and dissolve in 50 mls water in a 100 mil glass stoppered flask, add 3 mls hydrochloric acid and 2 gm. potassium iodide. Carefully stopper the flask and heat for half an hour at 40° C. After cooling titrate with decinormal sodium thiosulphate until the color is just discharged.

147. Calculation of Analysis.

Ferric salts, when added to a solution of potassium iodide in the presence of hydrochloric acid, liberate an equivalent quantity of iodine, the reaction being as follows:



Supposing that it required 14.2 mls decinormal thiosulphate to decolorize the iodine liberated by 0.31 gm. ferric chloride, then

1 mil decinormal thiosulphate = 1 mil decinormal iodine.

14.2 mls decinormal thiosulphate = 14.2 mls decinormal iodine.

1 mil decinormal iodine = 1 mil decinormal ferric chloride.

14.2 mls decinormal iodine = 14.2 mls decinormal ferric chloride.

1 mil decinormal ferric chloride = .016222 gm. ferric chloride.

14.2 mls decinormal ferric chloride = $14.2 \times .016222$ or .23035 gm. ferric chloride.

The .31 gm. of crystallized ferric chloride therefore contains .23035 gm. of true ferric chloride or 74.3%.

Calculate the percentage of metallic iron it contains.

148. Estimation of Arsenous Acid in Fowler's Solution

Accurately weigh 20 mls of Fowler's Solution, dilute with 75 mls of distilled water, acidulate very slightly with dilute HCl, and dissolve in the solution 2 gm. sodium bicarbonate. Add a few drops of starch solution and add decinormal iodine solution from a burette until a blue color is produced.

149. Calculation of Analysis.

The potassium arsenite is oxidized to arsenate by the iodine. The following equation indicates the manner in which arsenous anhydride is oxidized to arsenic anhydride:



Since 4×126.92 parts of iodine are necessary for the oxidation of 197.92 parts of arsenous oxide, 126.92 parts of the former will oxidize 49.48 parts of the latter.

Supposing 20 mls of decinormal iodine were required for the oxidation of the As_2O_3 in the 10 mls of Fowler's solution and

$$\begin{aligned} 1 \text{ mil decinormal iodine} &= 1 \text{ mil decinormal } \text{As}_2\text{O}_3. \\ 20 \text{ mls decinormal iodine} &= 20 \text{ mls decinormal } \text{As}_2\text{O}_3. \\ 1 \text{ mil decinormal } \text{As}_2\text{O}_3 &= .004948 \text{ gm. } \text{As}_2\text{O}_3. \\ 20 \text{ mls decinormal } \text{As}_2\text{O}_3 &= .09896 \text{ gm. } \text{As}_2\text{O}_3. \end{aligned}$$

The 10 mls of Fowler's solution therefore contained .09896 gm. of arsenous oxide or .9896%.

150. Preparation of Decinormal Bromine Solution.

$$1 \text{ Liter} = 7.992 \text{ gm. Bromine.}$$

Dissolve 3.2 gm. of potassium bromate and 50 gm. potassium bromide in sufficient water to measure 900 mls.

Place exactly 20 mls of this solution in a 250 ml glass stoppered flask, add 75 mls water and 5 mls pure hydrochloric acid, and immediately insert the stopper. After shaking a few times, quickly introduce 5 mls potassium iodide solution, being careful to prevent the escape of bromine vapors. Agitate

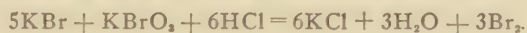
again, then add from the burette decinormal sodium thiosulphate until the color due to the iodine is just discharged, a few drops of starch solution being added toward the end of the reaction.

Note the number of mls of decinormal thiosulphate used and then dilute the bromine solution so that equal volumes of it and of decinormal thiosulphate will exactly correspond to each other under the above conditions.

Assuming that the 20 mls of bromine solution required 26.4 mls decinormal sodium thiosulphate to completely discharge the iodine tint, then each 20 mls of bromine solution must be diluted to measure 26.4 mls.

Owing to the volatility of bromine it is not practical to use a solution of free bromine, but instead the above solution from which a definite amount of bromine is liberated when wanted on the addition of HCl.

The following reaction takes place on the addition of hydrochloric acid to decinormal bromine solution:



151. Valuation of Phenol.

Weigh carefully from one to two grams of phenol and dissolve in 1000 mls of water and after thoroughly mixing, place 25 mls in a glass stoppered flask of about 200 mls capacity, add 30 mls decinormal bromine solution and 5 mls hydrochloric acid. Immediately insert the stopper and shake repeatedly during half an hour, then remove the stopper just sufficiently to introduce 5 mls of a 20% solution of potassium iodide, being careful that no bromine vapor escapes. Shake thoroughly and titrate with decinormal sodium thiosulphate, adding a few drops of starch solution toward the end of the reaction, until the blue color is just discharged.

152. Calculation of Analysis

In the presence of an excess of bromide phenol combines with it to form the insoluble tribrom-phenol, thus:



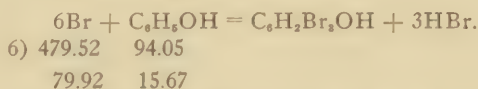
An excess of bromine being necessary, more of the decinormal bromine solution is added than is required to convert all the phenol into tribrom-phenol, and the excess is determined and deducted from the amount used, the difference being the amount taken up by the phenol.

The bromine excess is determined by adding potassium iodide from which each atom of bromine liberates one atom of iodine, the iodine being then determined in the usual manner by decinormal thiosulphate.

On adding the KI solution the following reaction takes place:



Assuming that in the titration of phenol 6 mils of decinormal thiosulphate were required to discharge the color, 1 mil decinormal thiosulphate being equal to 1 mil decinormal bromine, and 30 mils decinormal having been used, then $30 - 6 = 24$, the number of mils decinormal bromine required by the phenol. According to the equation



79.92 parts of bromine combine with 15.67 parts of phenol, and as 1 mil decinormal bromine contains .007992 bromine, it will equal .001567 phenol and 24 mils will equal $24 \times .001567$ or .037608 gm. phenol.

If the phenol dissolved in the 1000 mils of water weighed 1.9 gm., 25 mils of this solution contained .0475 gm., which was the amount used. .0475 gm. of the phenol tested contained .037608 pure phenol, or 79.1%.

PART SIX.

GRAVIMETRIC ANALYSIS.

153. *Estimation of Water of Crystallization.*

Heat to redness a porcelain crucible, allow to cool in desiccator and weigh. Place from one to two grams of crystallized and uneffloresced sodium carbonate in the crucible and weigh again. Gently heat the crucible and its contents, in order to drive off the water. When the latter appears to have been all volatilized, replace the lid and heat to low redness, cool in desiccator and weigh. Repeat the heating, cool and weigh again. There should be no decrease in weight, showing that all the water was removed in the first operation. Should there be a decrease, however, repeat the heating until the weight is constant.

Ascertain the amount of water driven off and calculate the percentage, comparing it with the calculated percentage obtained from the formula for crystallized sodium carbonate, $\text{Na}_2\text{CO}_3 + 10\text{H}_2\text{O}$.

154. *Estimation of Iron in Organic Salts.*

Into a recently ignited and weighed porcelain crucible place about one gram of ferric citrate, and weigh accurately. Cover the crucible and apply the flame cautiously until vapors cease to be evolved, remove the lid, slightly incline the crucible and expose to a red heat until all carbonaceous matter has been consumed. Cool in desiccator and weigh. The residue is ferric oxide, Fe_2O_3 . From the weight of the ferric oxide obtained, calculate the amount of metallic iron in the ferric citrate and from this the percentage.

The iron in iron and potassium tartrate may be estimated

in the same way, except that in this case the ferric oxide must be thoroughly washed in order to remove the potassium carbonate formed during the incineration, and again ignited before weighing.

155. Estimation of Iron by Precipitation as Hydroxide.

Weigh a small bottle containing solution of ferric chloride, pour a little of the solution into a medium sized beaker and weigh the bottle again. The difference will give the amount of ferric chloride solution taken. Dilute the contents of the beaker with 100 mls of water, boil gently; pour in an excess of ammonia water, boil again until the odor of ammonia is no longer noticeable (adding water if necessary), and set aside until the ferric hydroxide has deposited. Pour the supernatant liquid through a plain filter of known ash content; wash the precipitate three or four times by decantation with boiling water; transfer the precipitate to the filter, using a glass rod with a small piece of rubber tubing on the lower end to remove the precipitate completely from the beaker; wash the filter and precipitate until the filtrate no longer yields a cloudiness with silver nitrate and dry the precipitate on the filter. When nearly dry, the precipitate and filter are rolled up and placed in a porcelain crucible which has been recently ignited and weighed, covered, and gently heated until the vapors of burning paper appear; the heat is now increased for a few moments, the cover removed, and the crucible inclined and strongly heated until all the carbon is consumed. Replace the cover, heat to redness, cool in desiccator and weigh. The weight of the ferric oxide is obtained by subtracting the weight of the crucible and filter ash. Calculate the percentage of metallic iron contained in the solution of ferric chloride.

156. Estimation of Arsenic as Arsenous Sulphide.

Weigh accurately about .5 gm. of arsenous acid, dissolve with the aid of heat in 100 mls of distilled water containing 3 mls hydrochloric acid. Through the hot solution pass hydrogen sulphide gas until it acquires a strong odor of the latter.

Remove the excess of gas by heating and passing a current of carbon dioxide through it. Collect the sulphide on a dried and weighed filter, wash thoroughly, dry at 100° C., cool in desiccator, and weigh.

From the weight of As_2S_3 calculate the weight of arsenic.

157. Estimation of Mercury as Mercuric Sulphide.

Weigh accurately about .5 gm. of mercuric chloride and dissolve in 100 mls of distilled water. Pass hydrogen sulphide gas through the solution until thoroughly saturated. Collect the precipitate on a dried and weighed filter, wash first with water, then with absolute alcohol, and finally, to remove any free sulphur, with a mixture of equal parts of ether and carbon disulphide. Dry at 100° C., cool in desiccator, and weigh.

From the weight of HgS found calculate the weight of mercury in the mercuric chloride taken.

158. Estimation of Zinc as Oxide.

Weigh accurately about .7 gm. of pure crystallized zinc sulphate ($\text{ZnSO}_4 + 7\text{H}_2\text{O}$), dissolve in about 100 mls of water, heat to boiling and precipitate with sodium carbonate solution, continuing the boiling for some minutes. Allow the precipitate to settle, decant through a filter of known ash content, wash twice with boiling water by decantation, transfer to the filter, wash, and dry. Introduce the filter and contents into a porcelain crucible, previously ignited and weighed, heat gently for a short time and then for some time at a bright red heat, cool in desiccator and weigh. The ignition changes the zinc carbonate to oxide.

From the ZnO found calculate the weight of zinc.

159. Estimation of Aluminum as Oxide.

Dissolve about 1 gm. of pure alum, accurately weighed, in 200 mls of water, add a slight excess of ammonium hydroxide and boil until only a slight odor of ammonia is perceptible. Collect the precipitated aluminum hydroxide on a filter of

known ash content, wash thoroughly with boiling water, and dry. Transfer the dry filter and contents to a previously ignited and weighed crucible, heat, first gently, then to a bright red heat for some time, allow to cool in desiccator, and weigh.

From the weight of Al_2O_3 found calculate the weight of aluminum in the alum taken.

160. *Estimation of Calcium as Carbonate.*

Weigh accurately about .5 gm. of precipitated calcium carbonate, dissolve in dilute hydrochloric acid, in a tall beaker, add 10 mls ammonium chloride solution, and make alkaline by ammonium hydroxide. Now add ammonium oxalate solution in excess and heat, keeping just below the boiling point until the precipitate aggregates. Collect the precipitate on a filter, wash with boiling water until the filtrate is free from chlorides, and dry at 100°C .

Transfer the precipitate carefully from the filter into a crucible previously ignited and weighed, and ignite gently. Moisten with a solution of pure ammonium carbonate, and heat until no more fumes are evolved, being careful to avoid a temperature sufficiently high to decompose the calcium carbonate, cool in desiccator, and weigh.

161. *Estimation of Silicic Acid.*

(a) *In Soluble Silicates.*

Accurately weigh about 2 gm. solution of sodium silicate, dissolve in 100 mls water and add a slight excess of hydrochloric acid. Evaporate the solution to dryness on a water-bath, being careful to break up all lumps formed during the drying. Dry the residue at 120°C . for an hour. After cooling moisten the mass with concentrated hydrochloric acid, add water, and boil. The insoluble residue of silica (SiO_2), is collected on a filter of known ash content, washed, dried and transferred to a previously ignited and weighed crucible, ignited, cooled, and weighed.

(b) In Insoluble Silicates.

Insoluble silicates must be decomposed by mixing a weighed quantity of the finely powdered substance with four times its weight of sodium potassium carbonate fusing mixture and fusing the whole in a platinum crucible for half an hour. The crucible must be well covered during fusion. After cooling the fused mass is dissolved in warm dilute hydrochloric acid, evaporated to dryness, and treated as above described at 120°C. , etc.

162. Determination of Sulphuric Acid as Barium Sulphate.

Weigh on a watch glass accurately about .5 gm. potassium sulphate, dissolve in a large beaker in 150 mls water, rinsing the watch glass with some of the water, add 5 mls hydrochloric acid and heat to boiling. To the boiling solution add a slight excess of a hot solution of barium chloride previously acidulated with hydrochloric acid, keep the liquid near the boiling point for five minutes and allow to settle.

Decant the supernatant liquid through a plain filter of known ash content, wash the precipitate several times by decantation with hot water and transfer it to the filter. Wash thoroughly with boiling water until free from chlorides and drain. While still moist the filter and contents are placed in a previously ignited and weighed porcelain crucible, covered and gently heated until the paper is burnt; the cover is now removed and the crucible and contents heated until the residue is white. Replace the cover, heat to redness, cool in desiccator and weigh. The precipitate is barium sulphate. From the amount of barium sulphate obtained calculate the percentage of potassium sulphate in the sample used.

163. U. S. P. Test for Heavy Metals.

Chemicals.—This test is to be used to detect the presence of undesirable metallic impurities in official chemical substances or their solutions; these must not respond affirmatively within the stated time. Acidulate 10 mls of a solution of the substance in distilled water (1 in 50), contained in a test tube of about 40 mls capacity and of about 2.5 cm. diameter, with

1 mil of diluted hydrochloric acid (unless otherwise directed),* warm it to about 50° C., add an equal volume of freshly prepared hydrogen sulphide T.S., stopper, and allow the mixture to stand at 35° C. for half an hour. At the end of this time the mixture should still possess the odor of hydrogen sulphide; if not, it should be thoroughly saturated with the gas and again set aside for half an hour. The color produced, if any, is not greater than that observed by a blank test made in the same manner and with the same quantities of the reagents (omitting the solution to be tested), the solutions being viewed crosswise by reflected light while held against a white surface. A slight turbidity due to separation of sulphur from the hydrogen sulphide may occur.

Volatile Oils.—Shake 10 mils of the oil with an equal volume of distilled water to which a drop of hydrochloric acid has been added and pass hydrogen sulphide through the mixture until it is saturated; no darkening in color will be produced in either the oil or the water (lead or copper).

164. *Determination of Alcohol in Official Preparations.*

Place 25 mils of the preparation, measured at any definite temperature between 15° and 30° C., in a distilling flask of about 200 mils capacity, provided with a high side tube connected with a suitable condenser; add 50 mils of distilled water, distil into a 50 mil graduated flask, at such a rate that about 48 mils of distillate will be received in a half hour; if volatile products other than alcohol are known to be absent, bring to the original temperature and dilute with water of the same temperature to 50 mils.

Determine the specific gravity of the distillate at any definite temperature between 15° and 30° C. by means of a pycnometer, and find the per cent. of absolute alcohol by volume by reference to the alcoholometric table, and multiply by the dilution factor to find the per cent. of alcohol in the original preparation.

* First neutralize solutions of acids carefully by the addition of ammonia water and then acidify as above directed.

If the preparation is suspected to contain less than 25 per cent. of alcohol, 50 mils should be taken for analysis; if it is suspected to contain over 50 per cent. of alcohol, 12.5 mils should be taken for analysis. In either case, the volume of the distillate should be made up as usual to 50 mils.

If the preparation contains iodine, combine it with zinc dust. (Sodium thiosulphate may be used instead of zinc dust, in which case several drops of sodium hydroxide T.S. should be added to prevent sulphur from distilling over.)

If volatile acids are present, neutralize with sodium hydroxide T.S.; if volatile bases are present, neutralize with dilute sulphuric acid; if both volatile acids and volatile bases are present, neutralize first with dilute sulphuric acid and distil about 50 mils, neutralize this distillate with sodium hydroxide T.S., and distil once more to obtain in the usual manner 50 mils.

If acetone, camphor, chloroform, ether, glycerin (30 per cent. or over), volatile oils or other volatile products are known or suspected to be present, transfer the first distillate to a separatory funnel and saturate it with sodium chloride, add 15 mils of petroleum benzin (boiling point 40°–50° C.) and shake the mixture for one or two minutes. After the liquids have *completely* separated, draw off the lower alcoholic salt solution, with a second separator, and repeat the extraction with 15 mils of petroleum benzin; draw off the lower alcoholic salt solution into a distilling flask of about 200 mils capacity, wash the combined petroleum benzin solutions with about 25 mils of saturated sodium chloride solution and add the washings to the distilling flask. Distil into a 50 mil graduated flask at such a rate that about 45 to 48 mils of distillate are received within a half hour. Bring to the original temperature, dilute with water of the same temperature to 50 mils, and determine the specific gravity and alcoholic per cent. as directed above.

165. *Determination of Ash or Non-volatile Matter.*

Chemicals.—Weigh accurately from 1 to 2 gm. of the chemical or the amount directed in the text, and place it in a

tared platinum or porcelain crucible. Ignite it, at first very gently, and then gradually raise the temperature until the carbon, if formed, is completely burned off. Conduct the ignition in a place protected from air currents and use as low a temperature as possible to effect the combustion of the carbon. When the carbon has completely disappeared, transfer the crucible while still hot to a desiccator and weigh it when cold. In the case of substances which increase in volume on ignition, or where it is very difficult to burn off the carbon, the determination is facilitated by using a platinum dish of suitable size, or by igniting the weighed quantity of the substance, in the manner described above, in several separate portions.

To test for non-volatile matter in volatile inorganic chemicals proceed as directed above, using as low a temperature as possible.

Vegetable Drugs.—Incinerate 2 gm. of the ground drug until free from carbon and of constant weight, using a tared crucible and the lowest temperature possible, then determine the weight of the ash. If a carbon-free ash cannot be obtained in this way, exhaust the charred mass with distilled water, collect the insoluble residue on a filter paper of known ash content, incinerate the residue and filter paper until the ash is white or nearly so, then add the filtrate, evaporate it to dryness and heat the whole to a low redness. Finally determine the weight of the ash, deducting the weight of the ash from the filter.

PART SEVEN.

PHARMACEUTICAL ASSAYING.

166. Assay of Fluidextract of Guarana.

Fluidextract of Guarana 5 Mils.
Chloroform,
Ammonia Water,
Iodine Test Solution,
Distilled Water, of each, a sufficient quantity.

Transfer, by means of a graduated pipette, to a separator 5 mils of fluidextract of guarana, and add 1 mil of ammonia water and 15 mils of chloroform. Shake the mixture and, when the liquids have separated, draw off the chloroform solution into a beaker. Wash the liquid remaining in the separator with two successive portions of chloroform of 10 mils each, and add the chloroform washings to the beaker.

Test a portion of the liquid remaining in the separator with a few drops of iodine test solution, and if a precipitate is produced, wash the liquid in the separator with an additional 10 mils of chloroform.

Evaporate the mixed chloroform solutions, on a water-bath, to dryness and dissolve the solid residue in 20 mils of distilled water with the aid of heat. After cooling, filter the solution into a clean separator and wash the beaker and filter with several small portions of distilled water, adding the rinsings to the liquid in the separator.

Add 15 mils of chloroform to the liquid in the separator, shake, and after separation draw off the chloroform into a tared beaker. Repeat the washings with portions of chloroform of 10 mils each until the liquid in the separator no longer gives a precipitate with iodine test solution.

Evaporate the mixed chloroform solutions at 80° C. until the beaker ceases to lose weight. The weight of the dry residue represents the alkaloid in 5 mils of fluidextract of guarana.

167. Assay of Opium.

Opium in any condition to be valued ... 8 Gm.
Lime, freshly slaked,
Ammonium Chloride,
Alcohol,
Ether,
Tenth-normal Sulphuric Acid,
Fiftieth-normal Potassium Hydroxide,
Cochineal, Test Solution,
Distilled Water, of each, a sufficient quantity.

Introduce the opium, which, if fresh, should be in very small pieces, and if dry, in fine powder, into an Erlenmeyer flask having a capacity of about 250 mls. Add 80 mls of distilled water, stopper the flask with a sound cork and shake vigorously every ten minutes during three hours.

Pour the contents of the flask, as evenly as possible, upon a wetted filter having a diameter of not over 12 cm. and when the liquid has drained off, wash the residue with distilled water carefully dropped upon the edges of the filter and its contents until 120 mls of filtrate have been obtained.

Transfer the moist opium to a mortar by means of a spatula, rub to a smooth paste and then wash it back into the original flask with 50 mls of distilled water. Agitate thoroughly and return the whole to the filter.

When the liquid has drained off, wash the residue on the filter with 75 mls of distilled water. Evaporate the mixed filtrate and washings to about 40 gm., on a water-bath, transfer the liquid to a 50 ml graduated flask and wash the evaporating dish with sufficient distilled water to make the entire volume measure exactly 50 mls when cooled to room temperature.

Place in a small mortar 4 gm. of freshly slaked lime, add about 10 mls of the opium extract and rub to a smooth paste, then add the remainder of the extract and rinse the flask with exactly 10 mls of distilled water, adding the rinsings to the mortar, and stir frequently during 15 minutes, avoiding unnecessary loss by evaporation.

Filter through a dry filter about 10 cm. in diameter, and transfer exactly 30 mls of the filtrate (representing 4 gm. of opium) to an Erlenmeyer flask of suitable capacity. Add 2 mls of alcohol and 15 mls of ether, and after shaking add 1 gm.

of ammonium chloride. Stopper the flask and shake frequently during half an hour, then set aside in a cool place for 12 hours or over night to permit complete crystallization of the morphine.

Remove the stopper and brush any adhering crystals back into the flask. Decant the ethereal layer into a small funnel the neck of which is closed with a pledget of purified cotton. Rinse the flask and contents with 15 mls of ether and when the ether has passed through, wash the funnel and cotton with a small quantity of ether and then pour the aqueous liquid into the funnel without trying to remove the crystals from the flask.

Wash the crystals in the flask and the contents of the funnel with distilled water, previously saturated with morphine alkaloid, until the washings are colorless. Then add a few drops of distilled water to replace the morphinated water, incline the edge of the funnel over the mouth of the flask and by means of a glass rod carefully transfer the cotton and adhering crystals to the flask.

Insert the funnel into the neck of the flask and wash the funnel with 20 mls of tenth-normal sulphuric acid, followed by 10 mls of distilled water, applied drop by drop around the edge of the funnel.

Remove the funnel, replace the cork and warm the flask gently, with agitation, until the crystals are dissolved.

Rinse the cork with distilled water and titrate the excess of acid in the flask with fiftieth-normal potassium hydroxide, using cochineal test solution as the indicator.

From the amount of fiftieth-normal potassium hydroxide used in this titration determine the amount of tenth-normal sulphuric acid consumed by the alkaloid.

Each mil of tenth-normal sulphuric acid consumed corresponds to 0.028516 gm. of anhydrous morphine.

168. *Assay of Cinchona.*

Cinchona in No. 40 powder 5 Gm.
Chloroform,
Ether,
Ammonia Water,
Half-normal Sulphuric Acid,
Alcohol,
Distilled Water, of each, a sufficient quantity.

Introduce the cinchona into a 500 mil flask and add 200 mls of a mixture of chloroform 1 volume and ether 2 volumes. Stopper the flask with a sound cork, shake well and allow to stand 10 minutes.

Add 5 mls of ammonia water, shake frequently for 1 hour and allow to stand for 8 or 10 hours.

Add 10 mls of distilled water, shake vigorously and when the drug has settled, carefully decant 160 mls of the clear solution (representing 4 gm. of cinchona), and filter the decanted solution through a pledget of purified cotton into a separator, and rinse both cylinder and cotton with ether.

Extract the alkaloid from the chloroform-ether solution in the separator by shaking repeatedly with half-normal sulphuric acid, using portions of 10 mls each.

Collect the acid solution in a second separator, add ammonia water until the solution is distinctly alkaline to litmus, and again completely extract the alkaloids by shaking out repeatedly with chloroform. Filter each portion of chloroform as it comes from the separator through a pledget of purified cotton into a tared flask and wash the funnel and cotton with chloroform.

Evaporate the chloroform on a water-bath, add 5 mls of alcohol to the residue and again evaporate. Repeat the evaporation with 5 mls of alcohol and dry at 100° C. until the weight becomes constant.

The weight of dry residue will be the amount of total alkaloids in 4 gm. of cinchona.

169. *Assay of Fluidextract of Belladonna Root.*

Fluidextract of Belladonna Root ... 10 Mils.
Ammonia Water,
Chloroform,
Half-normal Sulphuric Acid,
Tenth-normal Sulphuric Acid,
Fiftieth-normal Potassium Hydroxide,
Cochineal, Test Solution,
Distilled Water, of each, a sufficient quantity.

Introduce the fluidextract of belladonna root into a separator and add 10 mls of distilled water and 2 mls of ammonia water.

Completely extract the alkaloids by shaking out repeatedly with chloroform, added in portions of 10 mls each.

Collect the chloroform washings in a clean separator and extract the alkaloids from the chloroform solution by shaking out repeatedly with half-normal sulphuric acid, using portions of 10 mls each, until the alkaloids are completely removed.

Collect the acid washings in a fresh separator, add ammonia water until the solution is decidedly alkaline to litmus, and extract the alkaloids from this liquid by repeatedly shaking out with chloroform, using portions of 10 mls each.

Evaporate the combined chloroform washings to dryness, dissolve the solid residue in exactly 5 mls of tenth-normal sulphuric acid, and titrate the liquid with fiftieth-normal potassium hydroxide, using cochineal test solution as indicator.

From the amount of fiftieth-normal potassium hydroxide used in the titration, calculate the amount of tenth-normal sulphuric acid consumed by the free alkaloid. Each mil of tenth-normal sulphuric acid thus consumed corresponds to 28.92 mgms. of the alkaloids of belladonna root present in 10 mls of the fluidextract.

170. *Assay of Fluidextract of Nux Vomica.*

Proceed as directed for the assay of fluidextract of belladonna root, but using 10 mls of tenth-normal sulphuric acid for dissolving the alkaloidal residue instead of 5 mls.

Each mil of tenth-normal sulphuric acid consumed corresponds to 36.42 mgms. of the mixed alkaloids of nux vomica.

171. *Assay of Solution of Formaldehyde.*

Solution of Formaldehyde3 Mils.
Normal Potassium Hydroxide Solution,
Solution of Hydrogen Dioxide,
Normal Sulphuric Acid,
Distilled Water,
Litmus Test Solution, of each, a sufficient quantity.

Transfer the solution of formaldehyde to a tared flask containing 10 mls of distilled water, stopper tightly and weigh accurately.

Add to the flask 50 mls of normal potassium hydroxide and follow immediately, but slowly, through a small funnel, with 50 mls of solution of hydrogen dioxide, previously rendered neutral to litmus with potassium hydroxide.

Heat the flask cautiously on a water-bath for 5 minutes, shaking occasionally.

Allow the mixture to cool, rinse the funnel and sides of the flask with distilled water and allow to stand 30 minutes.

Titrate the liquid with normal sulphuric acid, using litmus test solution as indicator.

From the amount of normal sulphuric acid used in the titration, calculate the amount of normal potassium hydroxide consumed by the formic acid produced by oxidation of the formaldehyde.

Each mil of normal potassium hydroxide thus consumed corresponds to 0.03002 gm. of CH_2O .

172. *Assay of Spirit of Nitrous Ether.*

Spirit of Nitrous Ether 40 Mils.
Powdered Potassium Bicarbonate,
Alcohol,
Potassium Iodide Test Solution,
Diluted Sulphuric Acid, of each, a sufficient quantity.

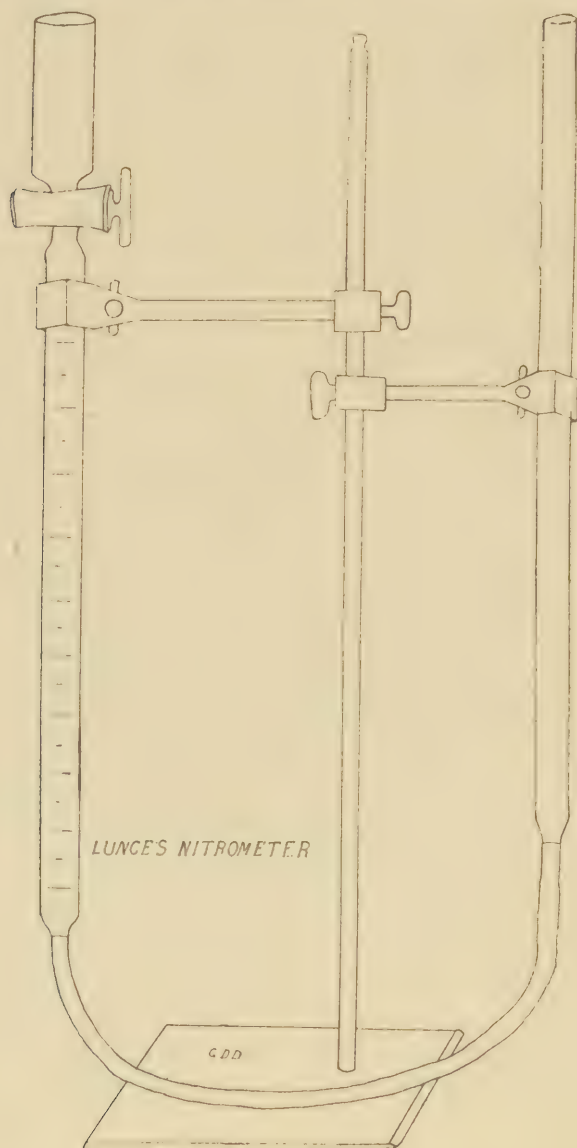
Shake the spirit of nitrous ether with 0.5 gm. of powdered potassium bicarbonate to neutralize free acid, decant the spirit into a tared 100 mil flask and weigh accurately.

Add sufficient alcohol to the flask to bring the volume to exactly 100 mls and thoroughly mix.

Introduce exactly 10 mls of the mixture into a nitrometer previously filled with saturated solution of common salt, follow it by 10 mls potassium iodide test solution, and afterwards by 5 mls diluted sulphuric acid.

Allow the nitrometer to stand until the volume of gas has become constant (30 to 60 minutes) and note the volume of gas collected.

Note the temperature and barometric readings in the laboratory, and correct the observed volume of gas to correspond to the



normal temperature of 25° C. and normal barometric pressure of 760 mm., using the table given in Part II of the U. S. P.

Multiply the corrected volume of gas in mls by 0.307 and divide the product by one-tenth of the weight in grams of the spirit of nitrous ether taken.

At standard temperature and pressure, the quotient represents the percentage of ethyl nitrite in the liquid.

173. Assay of Pepsin.

Pepsin0.1 Gm.
Coagulated Egg Albumen,
Normal Hydrochloric Acid,
Distilled Water, of each, a sufficient quantity.

Mix 25 mls of normal hydrochloric acid with 275 mls distilled water. This is known as the "acid liquid."

Dissolve the pepsin in 150 mls of the acid liquid. This is known as the "pepsin solution."

Immerse a hen's egg, not less than five nor more than twelve days old, in boiling water for 15 minutes. When sufficiently cooled, separate the coagulated albumen from the yolk and pellicle and rub it through a clean, dry No. 40 hair or brass sieve, rejecting the first portion that passes through the sieve.

Place 10 gm. of the succeeding portion of coagulated albumen in a wide-mouth bottle of 100 mls capacity, add 2 mls of the acid liquid and with the aid of a rubber-tipped glass rod moisten the albumen uniformly. Again add 2 mls of the acid liquid and gradually increasing portions of the acid liquid, manipulating all the time with the glass rod, until the total amount added measures 20 mls.

Thoroughly separate the particles of albumen from each other, rinse the rod with 15 mls more of the acid liquid and, after warming the mixture to 52° C., add exactly 5 mls of the pepsin solution.

Cork the bottle securely, invert it *three times*, and place it in a water-bath previously regulated to maintain a temperature of 52° C. Maintain this temperature for two and one-half hours, agitating the mixture every ten minutes by inverting the bottle *once*.

Transfer the albumen-pepsin mixture to a conical measure having an internal diameter not exceeding 1 cm. at the bottom, wash out the undigested albumen from the sides of the bottle with the aid of small portions of distilled water until the total amount used measures 50 mls. and add the liquid to the measure. Stir the mixture well and allow it to stand for half an hour.

If the deposit of undissolved albumen does not measure more than 1 mil, the pepsin employed is capable of digesting not less than 3000 times its own weight of albumen.

To ascertain the proteolytic power of pepsin stronger or weaker than the U. S. P. standard, make repeated trials exactly as above, using more or less of the pepsin solution, until the amount required to digest 10 gm. of albumen with not more than 1 mil of undigested residue is determined; then divide 15,000 by the quantity of pepsin solution expressed in mls. The quotient is the number of parts of coagulated albumen that one part of the pepsin will digest.

174. *Assay of Pancreatin.*

Pancreatin0.3 Gm.
Powdered Potato Starch,
Distilled Water,
Tenth-normal Iodine Solution, of each, a sufficient quantity.

Shake 10 gm. of powdered potato starch with about ten times its weight of cold distilled water, and after draining on a filter, wash with the same quantity of distilled water. Place the filter and starch in an air-bath and maintain a temperature of about 50° C. until the starch is sensibly dry. Reduce to a fine powder and preserve in a well stopped bottle.

Determine the percentage of water still remaining in the starch by drying about 0.5 gm. in an air-bath, gradually raising the temperature to 120° C., maintaining at that temperature for four hours and noting the loss of weight.

Of the washed and *partially dried* starch mix a quantity equivalent to 7.5 gm. of dried starch in a 400 mil beaker with 10 mls of cold distilled water, add 190 mls of boiling distilled water

and boil with constant stirring, for about five minutes, or until a translucent, uniform paste is produced.

Replace the water lost by evaporation and cool the paste to 40° C. in a water-bath previously adjusted to maintain this temperature, and add a solution of the pancreatin in 10 mls of distilled water just previously made at 40° C. Mix well and maintain the temperature of 40° C. for exactly five minutes, when a thin, nearly clear liquid is produced.

At once, add 0.1 mil of this liquid to a previously made mixture of 0.2 mil of tenth-normal iodine and 60 mls of distilled water. No blue, red or violet color should be produced, showing that the pancreatin has the starch digesting power required by the U. S. P.

A blue, red or violet reaction would show that part of the starch had not been converted, and hence that the pancreatin was below the U. S. P. standard.

162

APR 22 1919

QV 704 K21s 1918

10211890R



NLM 05070976 8

NATIONAL LIBRARY OF MEDICINE